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FINAL SAMPLING AND ANALYSIS PLAN REMEDIAL INVESTIGATION RCRA FACILITY INVESTIGATION SITE 35 DRMO SALVAGE YARD MCRD PARRIS ISLAND SC
07/01/2014
RESOLUTION CONSULTANTS

FINAL
SAMPLING AND ANALYSIS PLAN

REMEDIAL INVESTIGATION/RCRA FACILITY INVESTIGATION
SITE 35, DRMO SALVAGE YARD
MCRD PARRIS ISLAND, SOUTH CAROLINA

Revision: 1

Prepared For:

Department of the Navy
Naval Facilities Engineering Command Southeast
Building 135 N, Ajax Street
Jacksonville, Florida 32212-0030

Prepared By:

Resolution Consultants
A Joint Venture of AECOM & EnSafe
1500 Wells Fargo Building
440 Monticello Avenue
Norfolk, Virginia 23510

Contract Number: N62470-11-D-8013
CTO JM03

July 2014

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SAP WORKSHEET #1: TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN

REMEDIAL INVESTIGATION/RCRA FACILITY INVESTIGATION
SITE 35, DRMO SALVAGE YARD
MCRD PARRIS ISLAND, SOUTH CAROLINA

Revision: 0

Prepared For:

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Naval Facilities Engineering Command Southeast
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CTO JM03

May 2014

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Daniel Owens, NAVFAC Southeast
Remedial Project Manager/Date

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SAP WORKSHEET #1: TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN

LIMITED REMEDIAL INVESTIGATION/RCRA FACILITY INVESTIGATION
SITE 35, DRMO SALVAGE YARD
MCRD PARRIS ISLAND, SOUTH CAROLINA

Revision: 0

Prepared For:

Department of the Navy
Naval Facilities Engineering Command Southeast
Building 135 N, Ajax Street
Jacksonville, Florida 32212-0030

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CTO JM03

10 April 2013

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David Warren, Resolution Consultants Task Order Manager/Date	Daniel Owens, NAVFAC Southeast Remedial Project Manager/Date

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Executive Summary

Resolution Consultants has prepared this Uniform Federal Policy Sampling and Analysis Plan (SAP) for the Remedial Investigation (RI)/ Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) for Site 35, the Defense Reutilization and Marketing Office (DRMO) Salvage Yard at Marine Corps Recruit Depot (MCRD) Parris Island, under Contract No. N62470-11-D-8013, Comprehensive Long-term Environmental Action Navy (CLEAN), Contract Task Order (CTO) JM03.

Site 35, a 1.5-acre asphalt and concrete area in an industrial setting on unpopulated Horse Island, has operated as a DRMO salvage yard since 1964. The Site accepts recyclable materials, including paper, cardboard, wood, canvas, rubber, metals, spent ammunition casings, household appliances, electrical transformers, depleted fire extinguishers, and batteries. The DRMO also accepts hazardous materials or wastes deemed potentially useful or salvageable, including expired shelf-life, damaged, and off-specification paints, glazing compounds, dry cleaning solvents, cleaning compounds, petroleum products, batteries, mercury amalgam, and recovered silver pending resale or offsite disposal. Two 1,500-gallon aboveground storage tanks (ASTs) containing used motor oil with secondary containment and an emergency oil water separator, various paints and strippers, primarily stored in sealed 5-gallon buckets, and used motor oil and filters stored in 55-gallon drums were observed at the site in March 2012. No historical spills, releases, or incidents are documented at the Site.

Potential onsite sources of contamination include historical releases from debris, scrap metal, appliances, equipment, transformers, chemicals, and petroleum products. Concentrations of volatile organic compounds (VOCs), semi-volatile compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), and metals were detected above Ecological Screening Values (ESVs) per U.S. EPA Ecological Risk Assessment at Military Bases, dated December 22, 1998, and the U.S. EPA's current Protection of Groundwater Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites in historical surface soil samples collected from the top 6 inches at Site 35. Total chromium was detected above Industrial, Residential, and Protection of Groundwater RSLs and the ESV for hexalvalent chromium in two historical surface soil samples. Arsenic was detected above the Residential and Protection of Groundwater RSLs in a single, historical surface soil sample; however, the arsenic concentration is similar to its respective background level. The pesticide, 4,4-DDT, was detected above its respective U.S. EPA Residential RSL and Aroclor 1254, Aroclor 1260, and total PCBs were detected above both Residential and Industrial RSLs in historical surface soil samples collected at the Site. Based on available data, exceedances of Residential and Industrial RSLs are confined to surface soil in the vicinity of a storage area, historically used for temporary storage of various chemicals, petroleum products, batteries, and electrical transformers pending offsite disposal, and near the entry point of an earthen drainage ditch, which exits the Site to the southeast. No subsurface soil samples have been collected at the Site to date. Antimony was

detected slightly above the maximum contaminant level (MCL) in Site groundwater, which, to date, has only been tested for total analyte list metals (TAL) metals, cyanide, and tin.

Due to the limitations of the current dataset, impacts to environmental media resulting from historical operations at the Site cannot be fully evaluated at this time, thus preventing a decision on whether future action is required. This RI/RFI will fill data gaps associated with previous studies and assess potential threats to human health and the environment at the Site, through screening comparisons. Findings will provide information to support the development, evaluation, and selection of appropriate response alternatives, if necessary.

This SAP was generated for, and complies with, applicable United States Department of the Navy (Navy), United States Environmental Protection Agency (U.S. EPA) Region 4, and South Carolina Department of Health and Environmental Control (SCDHEC) requirements, regulations, guidance, and technical standards, as appropriate. This includes the Department of Defense (DoD), Department of Energy, and U.S. EPA Interagency Data Quality Task Force environmental requirements regarding federal facilities, as specified in the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) guidance (U.S. EPA, 2005) and the Navy's SAP guidance.

This SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the planned investigation at the DRMO Salvage Yard. Protocols for sample collection, handling, and storage, chain of custody, laboratory and field analyses, data validation, and reporting are also addressed. Field activities conducted under this SAP, which are scheduled to begin in December 2012, will be conducted in accordance with Resolution Consultants' Standard Operating Procedures and Site-Specific Health and Safety Plan, to be submitted under separate cover.

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List of Acronyms

°C	Degrees Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RSD	Relative standard deviation
bgs	Below Ground Surface
CAS	Chemical Abstracts Service
CCV	Continuing calibration verification
CCCs	Calibration check compounds
COD	Coefficient of the determination
CV	Calibration verification
CVAA	Cold vapor atomic absorption
CLEAN	Comprehensive Long-term Environmental Action Navy
COPC	Chemicals of potential concern
CSM	Conceptual Site Model
Cr VI	Hexavalent chromium
CTO	Contract Task Order
DDT	4,4'-Dichlorodiphenyltrichloroethane
DoD	Department of Defense
DoD QSM	Department of Defense Quality Systems Manual
DQO	Data Quality Objective
DRMO	Defense Reutilization and Marketing Office
DVM	Data Validation Manager
EICP	Extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
ESV	Ecological Screening Value
FOL	Field Operations Leader
GC	Gas chromatograph
GC/MS	Gas chromatograph/mass spectrometer
GC/ECD	Gas chromatography/electron capture detector
GCAL	Gulf Coast Analytical Laboratories
GPS	Global Positioning System
HASP	Health and Safety Plan
ICAL	Initial calibration
ICP	Inductively coupled plasma spectroscopy
ICP/MS	Inductively coupled plasma spectroscopy/mass spectrometer
ICV	Initial calibration verification
IDW	Investigation-Derived Waste
IRP	Installation Restoration Program
IS	Internal Standard

L	Liter
LCS	Laboratory Control Sample
LCS/LCSD	Laboratory Control Sample/laboratory control sample duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
MCL	Maximum Contaminant Level
MCRD	Marine Corps Recruit Depot
MD	Matrix Duplicate
µg/kg	Microgram per kilogram
µg/L	Microgram per liter
mL	Milliliter
MS	Mass Spectrometer
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NA	Not Applicable
NAVFAC SE	Naval Facilities Engineering Command Southeast
NIRIS	Naval Installation Restoration Information Solution
NTU	Nephelometric Turbidity Unit
oz	Ounce
PAH	Polynuclear Aromatic Hydrocarbon
PAL	Project Action Level
PCBs	Polychlorinated biphenyl compounds
PM	Program Manger
POC	Point of Contact
PRG	Preliminary Remediation Goal
PSQ	Principal study questions
PUPS	Palmetto Utility Protection Services
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
ORP	Oxidation reduction potential
QSM	Quality Systems Manual
r2	Correlation coefficient
RFA	Resource Conservation and Recovery Act Facility Assessment
RCRA	Resource Conservation and Recovery Act
RFs	Response factors
RI	Remedial Investigation
RI/RFI	Remedial Investigation/Resource Conservation and Recovery Act Facility Investigation
RL	Reporting limit
RPD	Relative Percent Difference
RPM	Remedial Project Manager

RRT	Relative retention times
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RT	Retention time
SAP	Sampling and Analysis Plan
SCDHEC	South Carolina Department of Environmental Control
SIM	Selective ion monitoring
SI	Site Inspection
SOP	Standard Operating Procedure
SSL	Soil Screening Level
SSO	Site Safety Officer
SVOC	Semi-volatile organic compound
SWMU	Solid waste management unit
TAL	Target analyte list
TCLP	Toxicity Characteristic Leaching Procedure
Tetra Tech	Tetra Tech NUS, Inc.
TOC	Top of Casing
TOM	Task Order Manager
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
U.S. EPA	United States Environmental Protection Agency
VOC	Volatile organic compound

SAP WORKSHEET #2: SAMPLING AND ANALYSIS PLAN IDENTIFYING INFORMATION

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Site 35 — Defense Reutilization and Marketing Office Salvage Yard, Marine Corps Recruit Depot (MCRD) Parris Island

Contractor Name: Resolution Consultants

Contract Number: N62470-11-D-8013

Contract Title: Comprehensive Long-term Environmental Action Navy (CLEAN)

Work Assignment Number: Contract Task Order (CTO) JM03

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

2. Identify regulatory program(s):
   - Comprehensive Environmental Response, Compensation, and Liability Act of 1980, as reauthorized by Superfund Amendments and Reauthorization Act
   - Resource Conservation and Recovery Act (RCRA), Title 42, United States Code 6901 et seq., 1976.

3. This document is a <u>project-specific</u> SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Data Quality Objective (DQO) Scoping Session — Partnering Team Meeting	24 April 2012
Regulatory Feedback — Partnering Team Meeting	12 February 2013

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

Title	Date
None	None

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

   Tenant — MCRP Parris Island

   Regulatory Oversight — U.S. EPA, Region 4

   Regulatory Oversight — South Carolina Department of Health & Environmental Control (SCDHEC)

7. Lead organization:
   Department of the Navy, Naval Facilities Engineering Command, Southeast (NAVFAC SE)

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

   All requirements are included in this SAP.

SAP WORKSHEET #3: DISTRIBUTION LIST

(UFP-QAPP Manual Section 2.3.2)

Distribution List				
SAP Recipients	**Title/Role**	**Organization**	**Telephone Number**	**E-Mail Address or Mailing Address**
Daniel Owens	Remedial Project Manager	Department of the Navy NAVFAC SE P.O. Box 30 Ajax Street, Bldg. 135N Jacksonville, Florida 32212-0030	904-542-6367	daniel.owens@navy.mil
Timothy Harrington	Installation Restoration Program Manager / Activity Point of Contact	MCRD Parris Island Building 172 Santo Domingo Street Parris Island, South Carolina 29905	843-228-3423	timothy.j.harrington@usmc.mil
Lila Llamas	Remedial Project Manager	U.S. EPA Region 4 Atlanta Federal Center 61 Forsyth Street, SW Atlanta, Georgia 30303-8960	404-562-9969	Llamas.Lila@epa.gov
Meredith Amick	Remedial Project Manager	SCDHEC Bureau of Land and Waste Division of Waste Management 2600 Bull Street Columbia, South Carolina 29201	803-898-0368	AmickMS@dhec.sc.gov
David Warren	Task Order Manager	Resolution Consultants 313 Wingo Way Mount Pleasant, South Carolina 29464	843-884-0029	dwarren@ensafe.com
Chad Tripp	Field Operations Leader and Site Safety Officer	Resolution Consultants 313 Wingo Way Mount Pleasant, South Carolina 29464	843-884-0029	ctripp@ensafe.com
Tina Cantwell	Project Quality Assurance Officer /Chemist	Resolution Consultants 5724 Summer Trees Drive Memphis, Tennessee 38134	901-937-4315	tcantwell@ensafe.com
Bonnie Capito	Administrative Record /Librarian	NAVFAC Atlantic 6506 Hampton Blvd Bldg. A Norfolk, Virginia 23508	757-322-4785	bonnie.capito@navy.mil

Distribution List				
SAP Recipients	**Title/Role**	**Organization**	**Telephone Number**	**E-Mail Address or Mailing Address**
Brenda Martinez	Laboratory Project Manager	Gulf Coast Analytical Laboratories 7979 GSRI Road Baton Rouge, Louisiana 70820	225-769-4900	brenda.martinez@gcal.com

Notes:
Each person listed in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization.

NAVFAC SE	=	Department of the Navy, Naval Facilities Engineering Command, Southeast
MCRD	=	Marine Corps Recruit Depot
SCDHEC	=	South Carolina Department of Environmental Control

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:

- 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.

- E-mails will be sent to the Navy, Resolution Consultants, and subcontractor project personnel who will be requested to verify by e-mail that they have read the applicable SAP/sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files, as 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.

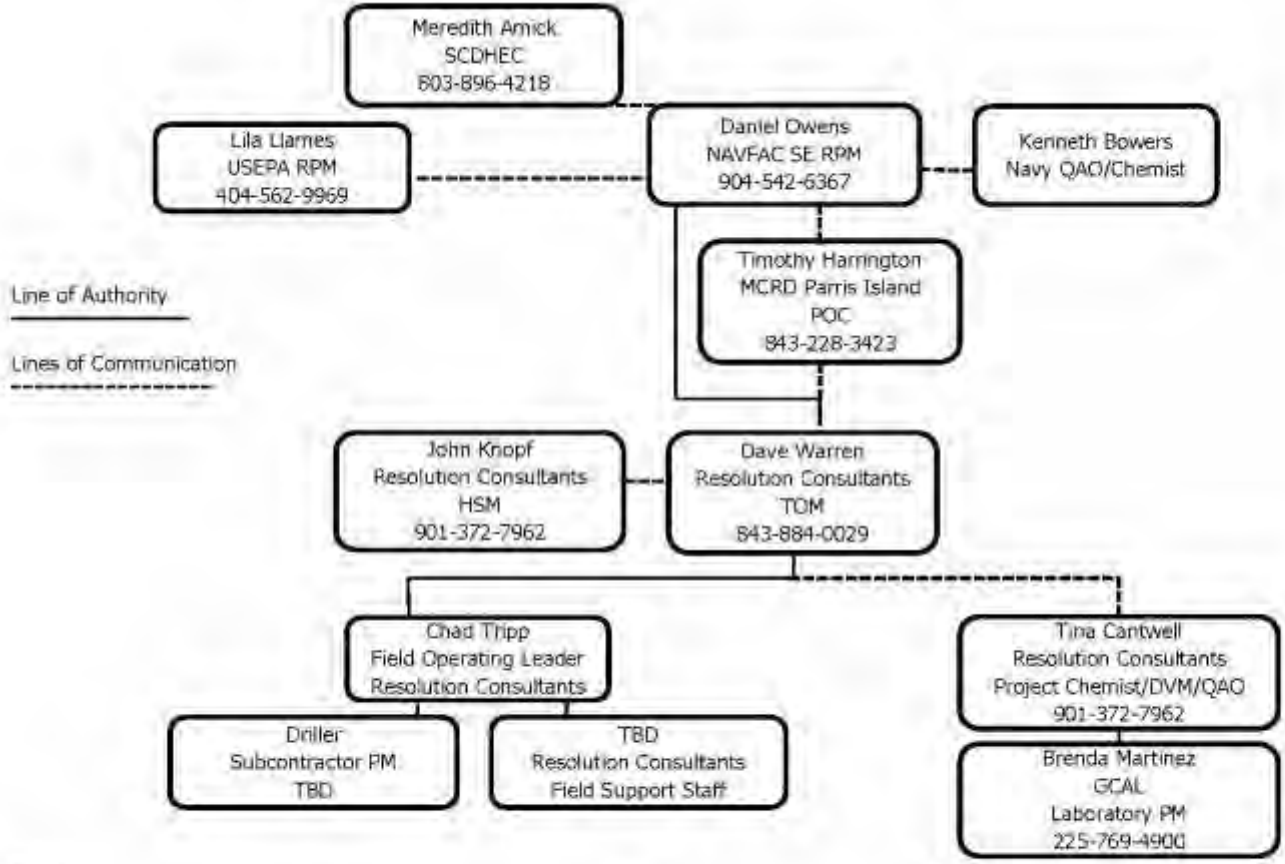
Project Personnel Sign-Off Sheet					
Name	**Organization/Title/Role**	**Telephone Number**	**Signature/E-Mail Receipt**	**SAP Section Reviewed**	**Date SAP Read**
Navy and Regulator Partnering Team Personnel					
Daniel Owens	Navy Remedial Project Manager (Manages project for the Navy)	904-542-6367		All	
Timothy Harrington	MCRD Parris Island Installation Restoration Program Manager (Activity Point of Contact)	843-228-3423		All	
Lila Llamas	U.S. EPA Remedial Project Manager (Provides Regulatory Oversight)	404-562-9969		All	
Meredith Amick	SDHEC Remedial Project Manager (Provides Regulatory Oversight)	803-896-4218		All	
Resolution Consultants' Partnering Team Personnel					
David Warren	Task Order Manager (Manages project for Resolution Consultants)	843-884-0029		All	
Chad Tripp	Field Operations Leader and Site Safety Officer(Oversees field activities and site safety)	843-884-0029		All	
Tina Cantwell	Quality Assurance Officer/Project Chemist (Oversees quality and subcontracted analytical services)	901-372-7962		All	
Subcontractor Personnel					
Brenda Martinez	Gulf Coast Analytical Laboratories/Laboratory Project Manager (Representative for subcontracted analytical services)	225-769-4900		Worksheets #6, #12, #14, #15, #19, #20, #23-28, #30, and #34-36	

Notes:

NAVFAC SE = Department of the Navy, Naval Facilities Engineering Command, Southeast
MCRD = Marine Corps Recruit Depot
SCDHEC = South Carolina Department of Environmental Control

SAP WORKSHEET #5: PROJECT ORGANIZATIONAL CHART

(UFP-QAPP Manual Section 2.4.1)



Notes:

DVM =	Data Validation Manager	GCAL =	Gulf Coast Analytical Laboratory	SCDHEC =	SC Depart of Health and Environmental Control
HSM =	Health & Safety Manager	RPM =	Remedial Project Manager	POC =	Point of Contact
PM =	Project Manager	QAO =	Quality Assurance Officer	TOM =	Task Order Manager
NAVFAC SE =	Naval Facility Engineering Command Southeast			USEPA =	U.S. Environmental Protection Agency

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SAP WORKSHEET #6: COMMUNICATION PATHWAYS

(UFP-QAPP Manual Section 2.4.2)

Communication pathways for the SAP are shown below.

Communication Pathways				
Communication Drivers	**Responsible Entity**	**Name**	**Phone Number**	**Procedure (Timing, Pathway To/From, etc.)**
Regulatory Agency Interface	U.S. EPA RPM SCDHEC RPM NAVFAC SE RPM	Lila Lllamas Meredith Amick Daniel Owens	404-562-9969 803-898-0368 904-542-6367	The NAVFAC SE RPM will contact each regulatory agency via phone and/or e-mail within 24 hours of recognizing the issue whenever issues arise.
Field Progress Reports	Resolution Consultants TOM Resolution Consultants FOL	David Warren Chad Tripp	843-884-0029 843-884-0029	The Resolution Consultants FOL will contact the Resolution Consultants TOM on a daily basis via phone, and every 1-2 days summarizing progress via e-mail.
Gaining Site Access	Resolution Consultants FOL MCRD Parris Island POC	Chad Tripp Timothy Harrington	843-884-0029 843-228-3423	The Resolution Consultants FOL will contact the MCRD Parris Island POC verbally or via e-mail at least 3 days prior to commencement of field work to arrange for access to the site for all field personnel.
Obtaining Utility Clearances for Intrusive Activities	Resolution Consultants FOL MCRD Parris Island POC	Chad Tripp Timothy Harrington	843-884-0029 843-228-3423	The Resolution Consultants FOL will coordinate verbally or via e-mail with MCRD Parris Island POC at least 14 days in advance of site access to initiate the utility clearance process for all intrusive sampling locations. The Resolution Consultants FOL will contact both the Palmetto Utility Protection service and MCRD Parris Island personnel (as directed by the MCRD Parris Island POC) verbally or via e-mail at least 14 days prior to commencement of field work to complete a utility clearance ticket for areas under investigation.
Stop Work Due to Safety Issues	Resolution Consultants TOM Resolution Consultants FOL Resolution Consultants QAO Resolution Consultants Health and Safety Manager NAVFAC SE RPM MCRD Parris Island POC	David Warren Chad Tripp Tina Cantwell John Knopf Daniel Owens Timothy Harrington	843-884-0029 843-884-0029 901-937-4315 901-367-7962 904-542-6367 843-228-3423	If Resolution Consultants is the responsible party for a stop work command, the Resolution Consultants SSO will inform onsite personnel, subcontractor(s), the MCRD Parris Island POC, and the identified Partnering Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must verbally inform the Resolution Consultants SSO within 15 minutes, and the Resolution Consultants SSO will then follow the procedure listed above.

Communication Pathways				
Communication Drivers	**Responsible Entity**	**Name**	**Phone Number**	**Procedure (Timing, Pathway To/From, etc.)**
SAP Changes Prior to Field/ Laboratory Work	Resolution Consultants FOL Resolution Consultants TOM NAVFAC SE RPM MCRD Parris Island POC U.S. EPA RPM SCDHEC RPM	Chad Tripp David Warren Daniel Owens Timothy Harrington Lila Llamas Meredith Amick	843-884-0029 843-884-0029 904-542-6367 843-228-3423 404-562-9969 803-898-0368	The Resolution Consultants TOM will document the proposed changes via a Field Task Modification Request form within 5 days and send the NAVFAC SE RPM a concurrence letter within 7 days of identifying the need for change if necessary. SAP amendments will be submitted by the Resolution Consultants TOM to the NAVFAC SE RPM and MCRD Parris Island POC for review and approval. The Navy RPM will notify the U.S. EPA RPM and the SCDHEC PRM of any required changes to the approved SAP. Any change affecting the scope or implementation of the sampling program will be made only upon authorization of the Navy RPM and regulatory agencies.
SAP Changes in the Field	Resolution Consultants TOM Resolution Consultants FOL NAVFAC SE RPM MCRD Parris Island POC U.S. EPA RPM SCDHEC RPM	David Warren Chad Tripp Daniel Owens Timothy Harrington Lila Llamas Meredith Amick	843-884-0029 843-884-0029 904-542-6367 843-228-3423 404-562-9969 803-898-0368	The Resolution Consultants FOL will verbally inform the Resolution Consultants TOM on the day that the issue is discovered. The Resolution Consultants TOM will inform the NAVFAC SE RPM and the MCRD Parris Island POC (verbally or via e-mail) within 1 business day of discovery. The NAVFAC SE RPM will issue a scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed. The Resolution Consultants TOM will document the change via an a Field Task Modification Request form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form. Any change affecting the scope or implementation of the sampling program will be made only upon authorization of the Navy RPM and regulatory agencies.
Field Corrective Actions	Resolution Consultants TOM Resolution Consultants QAO NAVFAC SE RPM	David Warren Tina Cantwell Daniel Owens	843-884-0029 901-937-4315 904-0542-6409	The Resolution Consultants QAO will notify the Resolution Consultants TOM verbally or by e-mail within one business day that the corrective action has been completed. The Resolution Consultants TOM will then notify the NAVFAC SE RPM (verbally or by e-mail) within 1 business day.

Communication Pathways				
Communication Drivers	**Responsible Entity**	**Name**	**Phone Number**	**Procedure (Timing, Pathway To/From, etc.)**
Sample Receipt Variances	Resolution Consultants TOM Resolution Consultants Chemist GCAL Laboratory PM	David Warren Tina Cantwell Brenda Martinez	843-884-0029 901-937-4315 225-769-4900	The Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants FOL immediately upon receipt of any chain of custody/sample receipt variances for clarification or direction from the Resolution Consultants FOL. The Resolution Consultants FOL will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day, if corrective action is required. The Resolution Consultants TOM will notify (verbally or via e-mail) the Laboratory PM and the Resolution Consultants FOL within 1 business day of any required corrective action.
Reporting Laboratory Quality Variances	Laboratory PM Resolution Consultants Project Chemist Resolution Consultants TOM Resolution Consultants FOL Laboratory QAO NAVFAC SE RPM NAVFAC QAO/Chemist	Brenda Martinez Tina Cantwell David Warren Chad Tripp Tina Cantwell Daniel Owens Kenneth Bowers	225-769-4900 901-937-4315 843-884-0029 843-884-0029 901-937-4315 904-0542-6409 757-322-8341	Any planned Standard Operating Procedure variances from the quality elements specified in the *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2 October 2011 are identified in Worksheet #23. The Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants Project Chemist of any variance from the quality limits identified in this SAP on the day that the variance becomes known. The Resolution Consultants Project Chemist will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day of the need for corrective action, if the variance is a significant issue. The Resolution Consultants TOM will notify (verbally or via e-mail) the Laboratory PM and the Resolution Consultants FOL and Project Chemist within 1 business day of any required corrective action. The Laboratory QAM will document all quality variances in the Case Narrative of the Analytical Laboratory Report. If there are significant data Quality or Nonuseable data issues the NAVFAC LANT QAO/Chemist will be contacted to ensure the issues do not have the potential to impact other Navy projects.
Reporting Concerns Involving Laboratory	Resolution Consultants Project Chemist Resolution Consultants TOM	Tina Cantwell David Warren	901-937-4315 843-884-0029	If reported analytical results are inconsistent with the planned details identified in this SAP, the Resolution Consultants Project Chemist will notify (verbally or via e-mail) the Resolution Consultants TOM within 1 business day of identifying a concern to determine if corrective action is needed.

Communication Pathways				
Communication Drivers	**Responsible Entity**	**Name**	**Phone Number**	**Procedure (Timing, Pathway To/From, etc.)**
	Resolution Consultants FOL GCAL Laboratory PM	Chad Tripp Brenda Martinez	843-884-0029 225-769-4900	The Resolution Consultants TOM will notify (verbally or via e-mail) the Laboratory PM and the Resolution Consultants FOL and Project Chemist within 1 business day of any required corrective action.
Notification of Non-Usable Data	GCAL Laboratory PM Resolution Consultants Project Chemist/DVM Resolution Consultants TOM NAVFAC SE RPM U.S. EPA RPM SCDHEC RPM	Brenda Martinez Tina Cantwell David Warren Daniel Owens Lila Lllamas Meredith Amick	225-769-4900 901-937-4315 843-884-0029 904-542-6367 404-562-9969 803-898-0368	If the laboratory determines that any data they have generated is non-usable, the Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants project chemist within 1 business day of when the issue is discovered. The Resolution Consultants project chemist will notify (verbally or via e-mail) Resolution Consultants TOM within 1 business day of the need for corrective action, if the non-usable data is a significant issue (i.e., critical sample data). Corrective action may include resampling and/or reanalyzing the effected samples. If a Resolution Consultants project chemist or data validator identifies non-usable data during the data validation process, the TOM will be notified verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has resulted in non-usable data. The Resolution Consultants TOM will take corrective action appropriate for the identified deficiency to ensure the project objectives are met. The Resolution Consultants TOM will notify (verbally or via email) the NAVFAC RPM on any problems with the laboratory or analysis that could significantly affect the usability of the data or project failures that impact the ability to complete the scope of work. The NAVFAC RPM, may at their discretion, contact the Navy project chemist for assistance in problem resolution. Such notification will be made within 1 business day of when the issue is discovered. The NAVFAC RPM will notify the U.S. EPA and SCDHEC when any significant corrective action is taken.

Communication Pathways				
Communication Drivers	**Responsible Entity**	**Name**	**Phone Number**	**Procedure (Timing, Pathway To/From, etc.)**
Analytical Corrective Actions and Reporting Data Validation Issues	GCAL Laboratory PM Resolution Consultants Project Chemist/DVM Resolution Consultants TOM NAVFAC SE RPM	Brenda Martinez Tina Cantwell David Warren Daniel Owens	225-769-4900 901-937-4315 843-884-0029 904-542-6367	The Laboratory PM will notify (verbally or via e-mail) the Resolution Consultants Project Chemist within 1 business day of when an issue related to laboratory data is discovered. The Resolution Consultants Project Chemist will notify (verbally or via e-mail) the DVM and the Resolution Consultants TOM within 1 business day. Resolution Consultants' DVM or Project Chemist will notify the Resolution Consultants TOM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Resolution Consultants TOM will verbally advise the NAVFAC SE RPM within 24 hours of notification from the Resolution Consultants DVM or Project Chemist. The NAVFAC SE RPM will take corrective action appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed mass spectrometer tune or initial calibration verification. Corrective actions may include a consult with the Navy QAO/ Chemist.
Data Validation Corrective Actions	Resolution Consultants DVM Resolution Consultants TOM	Tina Cantwell David Warren	901-937-4315 843-884-0029	See "Notification of Non-Usable Data" procedure above. If a Resolution Consultants Data Validator identifies non-usable data during the data validation process that requires corrective action, the Resolution Consultants TOM will coordinate with the Resolution Consultants DVM to take corrective action appropriate for the identified deficiency to ensure the project objectives are met. Corrective action may include resampling and/or reanalyzing the affected samples, as determined by the Resolution Consultants TOM.

Notes:

U.S. EPA = United States environmental Protection Agency
SCDHEC = South Carolina Department of Environmental Control
NAVFAC SE = Department of the Navy, Naval Facilities Engineering Command, Southeast
MCRD = Marine Corps Recruit Depot
GCAL = Gulf Coast Analytical Laboratories
POC = Point of contact
FOL = Field Operations Leader
RPM = Remedial Project Manager
SSO = Site Safety Office
TOM = Task Order Manger
PM = Project Manger
QAM = Quality Assurance Manager
SAP = Sampling and Analysis Plan
DVM = Data Validation Manager
QAO = Quality Assurance Officer

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SAP WORKSHEET #7: PERSONNEL RESPONSIBILITIES TABLE

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Daniel Owens	NAVFAC RPM/Manages project activities for the Navy	NAVFAC Southeast	Primary Point of Contact for the Navy. Oversees project implementation, including scoping, data review, and evaluation, on behalf of the Navy.
Timothy Harrington	MCRD Parris Island Point of Contact/Oversees onsite project activities	MCRD Parris Island	Point of Contact for base-specific activity. Oversees onsite activities.
Lila Llamas	RPM/Regulatory Support	U.S. EPA Region 4	Functions as primary U.S. EPA interface. Participates in scoping and data review/evaluation, and provides review and approval of project deliverables.
Meredith Amick	RPM/Regulatory Support	SCDHEC	Functions as primary SCDHEC interface. Participates in scoping and data review/evaluation, and provides review and approval of project deliverables.
Jeffrey James	Deputy Program Manager/Manages CLEAN program	Resolution Consultants	Oversees CLEAN program on behalf of Resolution Consultants
David Warren	Task Order Manager/Manages project on a daily basis	Resolution Consultants	Primary point of contact for Resolution Consultants. Oversees project implementation, including financials, schedule, and technical aspects.
Chad Tripp	Field Operations Leader/ Site Safety Officer/Manages field operations and oversees site activities to ensure safety requirements are met	Resolution Consultants	Supervises, coordinates, and performs field activities. Responsible for on-site project-specific health and safety training and monitoring site conditions. Details are included in Health and Safety Plan.
Tina Cantwell	Quality Assurance Officer/Oversees project quality assurance activities	Resolution Consultants	Oversees development of procedures, training, control checks, and process correction/improvement actions.
Tina Cantwell	Project Chemist/Data Validation Manager	Resolution Consultants	Participates in project scoping. Oversees quality aspects associated with chemical analysis are implemented, documented, and maintained. Manages data validation activities.
John Knopf	Health and Safety Manager/Oversees health and safety activities	Resolution Consultants	Oversees CLEAN Health and Safety Program.
Brenda Martinez	Laboratory Project Manager/Analytical Subcontractor	Gulf Coast Analytical Laboratories	Oversees quality and technical aspects related to subcontracted analytical services.

Notes:

NAVFAC = Naval Facilities Engineering Command
RPM = Remedial Project Manager
MCRD = Marine Corps Recruit Depot
U.S. EPA = United States Environment
SCDHEC = South Carolina Department of Environmental Control
CLEAN = Comprehensive Long-term Environmental Action Navy

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SAP WORKSHEET #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

(UFP-QAPP Manual Section 2.4.4)

There is no special personnel training required for the execution of this CTO, worksheet not applicable.

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SAP WORKSHEET #9: PROJECT SCOPING SESSION PARTICIPANTS SHEET

(UFP-QAPP Manual Section 2.5.1)

Project Name: RI/RFI **Projected Date(s) of Sampling**: Summer 2013 **Task Order Manager**: David Warren	**Site Name**: Site 35, DRMO Salvage Yard **Site Location**: MCRD Parris Island, South Carolina
Date of Session: 24 April 2012 (April 2012 Partnering Meeting) **Scoping Session Purpose:** Data Quality Objective (DQO) Development	

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Dave Warren	TOM	Resolution Consultants	843-884-0029	dwarren@ensafe.com	Project Management/DQO Facilitator
Charles Cook	Former RPM	NAVFAC SE	904-542-6409	charles.cook2@navy.mil	Former NAVFAC SE RPM
Lila Llamas	RPM	U.S. EPA Region 4	404-562-9969	Llamas.Lila@EPA.gov	U.S. EPA RPM
Meredith Amick	RPM	SCDHEC	803-898-0368	AmickMS@dhec.sc.gov	SCDHEC RPM
Lisa Donohoe	Former Activity Coordinator	MCRD Parris Island	843-228-2779	lisa.donohoe@usmc.mil	Former Activity POC
Annie Gerry	Geologist	SCDHEC	803-898-0359	Gerryam@dhec.sc.gov	Regulatory Oversight
Kent Krieg	Risk Assessor	SCDHEC	803-898-0255	Kriegkm@dhec.sc.gov	Regulatory Oversight
Pat Franklin	Facilitator	The Management Edge	727-588-9481	Pat.Franklin@mail.com	Partnering Meeting Facilitator

Notes:

RI/RFI	=	Remedial Investigation/Resource Conservation and Recovery Act Facility Investigation
DRMO	=	Defense Reutilization and Marketing Office
TOM	=	Task Order Manger
DQO	=	Data quality objective
RPM	=	Remedial Project Manager
NAVFAC SE	=	Naval Facilities Engineering Command, Southeast
U.S. EPA	=	United States Environmental Protection Agency
MCRD	=	Marine Corps Recruit Depot
POC	=	Point of contact

Comments/Decisions: Approximately 1 week prior to the scoping session, Resolution Consultants provided the Partnering Team with draft versions of SAP Worksheets 10 (Conceptual Site Model) and 11 (Data Quality Objectives) and a draft figure to present proposed sampling locations. Such materials were discussed by the Partnering Team. Concerns were noted with regards to where historical materials were stored at the Site and as to when the Site was first paved.

Action Items: To address questions regarding when Site 35 was paved, U.S. EPA requested that historical aerial photographs be distributed for review by the Partnering Team. MCRD Parris Island agreed to provide Resolution Consultants with additional information on the oil water separator and drainage systems/leach field at Site 35. SCDHEC requested that historical regulatory comments related to previous work at Site 35, dated 25 June 2004 and 23 April 2010, be considered during preparation of the draft work plan documents. Resolution Consultants will move forward with preparation of the draft work plan/SAP for the RI/RFI at Site 35, accordingly.

Consensus Decisions:

In general, the Partnering Team agreed with information and proposed strategy as presented in the draft SAP Worksheets. Per U.S. EPA request, it was decided that in addition to subsurface soil sampling and analysis, surface soil samples should be collected and analyzed in the previously excavated drainage area at the Site's southern boundary. Such samples are to be collected outside and downgradient of small openings which allow drainage to exit the bermed pad that covers the majority of the Site. If results from surface samples taken directly outside and downgradient of the openings in the berm indicate the surface fill has been impacted, then additional sampling may be necessary to delineate contamination. Since detailed information on the historically used storage areas and when the Site was first paved is unavailable, it was decided that surface and subsurface samples should be collected from five additional soil borings in the northern (previously unstudied) portion of the Site. This will provide sufficient data across the entire Site. It was decided that one surface and one subsurface soil sample should be collected and analyzed from an additional soil boring adjacent to an emergency oil water separator at the Site.

Any additional comments or concerns will be provided during the forthcoming review of the draft work plan/SAP.

Project Name: RI/RFI **Projected Date(s) of Sampling:** Summer 2013 **Task Order Manager:** David Warren	**Site Name:** Site 35, DRMO Salvage Yard **Site Location:** MCRD Parris Island, South Carolina
Date of Session: 12 February 2013 (Partnering Meeting) **Scoping Session Purpose:** Clarify Level of Investigation	

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
Dave Warren	TOM	Resolution Consultants	843-884-0029	dwarren@ensafe.com	Project Management/DQO Facilitator
Daniel Owens	RPM	NAVFAC SE	904-542-6367	daniel.owens@navy.mil	NAVFAC SE RPM

Project Name: RI/RFI **Projected Date(s) of Sampling:** Summer 2013 **Task Order Manager:** David Warren	**Site Name:** Site 35, DRMO Salvage Yard **Site Location:** MCRD Parris Island, South Carolina
Date of Session: 12 February 2013 (Partnering Meeting) **Scoping Session Purpose:** Clarify Level of Investigation	

Lila Llamas	RPM	U.S. EPA Region 4	404-562-9969	Llamas.Lila@EPA.gov	U.S. EPA RPM
Meredith Amick	RPM	SCDHEC	803-898-0368	AmickMS@dhec.sc.gov	SCDHEC RPM
Lisa Donohoe	Activity Coordinator	MCRD Parris Island	843-228-2779	lisa.donohoe@usmc.mil	Former Activity POC
Annie Gerry	Geologist	SCDHEC	803-898-0359	Gerryam@dhec.sc.gov	Regulatory Oversight
Kent Krieg	Risk Assessor	SCDHEC	803-898-0255	Kriegkm@dhec.sc.gov	Regulatory Oversight
Gayle Waldron	Facilitator	The Management Edge	727-588-9481	Gwaldron@mgtedge.com	Partnering Meeting Facilitator

Notes:

RI/RFI	=	Remedial Investigation/Resource Conservation and Recovery Act Facility Investigation
DRMO	=	Defense Reutilization and Marketing Office
TOM	=	Task Order Manger
DQO	=	Data quality objective
RPM	=	Remedial Project Manager
NAVFAC SE	=	Naval Facilities Engineering Command, Southeast
U.S. EPA	=	United States Environmental Protection Agency
MCRD	=	Marine Corps Recruit Depot
POC	=	Point of contact

Comments/Decisions: Resolution Consultants provided a project update to the MCRD Parris Island Partnering Team and requested clarification on the level of investigation necessary at the Site. U.S. EPA indicated that the decision to proceed to study under an RI/RFI framework is necessary to fully document limited soil removal activities historically conducted at the Site and since surface soil has been impacted above applicable screening guidelines.

Action Items: U.S. EPA agreed to provide additional information to clarify the need for an RI/RFI at the Site. In email correspondence to the MCRD Partnering Team, dated 14 February 2013, Ms. Lila Llamas (U.S. EPA Region 4 RPM) provided an informal memorandum to summarizing field activities and results associated with 2003 surface soil sampling at the earthen drainage ditch, which exits the Site to the southeast. Summary tables and figures, included with the informal memorandum, indicate that offsite surface soil has been impacted above applicable screening levels. Ms. Llamas' email correspondence specified that the earthen ditch, soil beneath the Site's paved storage area, and the low-lying area adjacent to the Site's southern fence line, be evaluated during the forthcoming RI/RFI.

Decisions:

Based on subsequent correspondence with the Navy, Resolution Consultants has incorporated 2003 soil sampling results, as provided by U.S. EPA, into this SAP for RI/RFI efforts at Site 35. It should be noted that results from 2003 sampling efforts have not been formally documented to date. Furthermore, the informal memorandum only includes summary tables and a rough sketch of surface soil sampling locations. Analytical reports and survey data are not available, thus some uncertainty exists.

SAP WORKSHEET #10: CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2)

This worksheet presents general background information and the conceptual site model (CSM), which describes potential contamination routes and possible exposure pathways to humans and ecological receptors. The CSM serves as the basis for developing the sampling and analysis program.

10.1 Introduction

An RI/RFI will be conducted at Site 35, the DRMO Salvage Yard (the Site), in the northwestern portion of MCRD Parris Island, South Carolina. Results generated during previous studies indicate that surface soil may have been be impacted by historical operations at the Site; however, the current data set is insufficient to support a decision concerning whether or not future action is required. This RI/RFI will investigate potential contamination and assess potential threats to human health and the environment through screening comparisons. Specifically, this RI/RFI will fill data gaps associated with the SI and evaluate soil and groundwater for chemicals of potential concern (COPCs) associated with historical operations at the Site. Findings will provide information to support the development, evaluation, and selection of appropriate response alternatives, if necessary.

10.2 Site Description

MCRD Parris Island (the Depot) is comprised of Parris Island, seven smaller named islands, numerous unnamed islands, and salt marshes/tidal creeks on the coast of South Carolina. Figure 10-1 depicts the Site on a United States Geological Survey topographic map. The Depot, approximately1.5 miles south of the Town of Port Royal and the city of Beaufort and 30 miles northeast of Savannah, Georgia, has operated as a recruit training facility for the U.S. Marine Corps since 1915. Development includes administrative buildings, training facilities, recruit and family housing, building and vehicle maintenance shops, and community facilities. The projected future land use at MCRD Parris Island is consistent with current.

Site 35 is situated in an industrial setting, adjacent to the south of Vieques Road, on unpopulated Horse Island (Figures 10-2 and 10-3). The closest mainlands are Parris Island, approximately 1 mile to the southeast, and Port Royal, approximately 1.5 miles to the north. The Site currently operates as a collection/storage area for various metals and plastic recyclables and a storage area for equipment and boats. Building 953, in the southeastern portion of the Site, serves as a 90 day RCRA storage unit for hazardous materials and petroleum products awaiting offsite disposal. This active facility is not included in this study and, thus, will require evaluation at the time of closure. Two 1,500-gallon

aboveground storage tanks (ASTs) (containing used motor oil) with secondary containment and an emergency oil water separator, various paints and strippers (primarily stored in sealed 5-gallon buckets), and used motor oil and filters stored in 55-gallon drums were observed at the site, during a March 2012 site visit by Resolution Consultants and the Navy. The ASTs remain in active use, storing motor oil awaiting offsite disposal.

10.3 Physical Setting

MCRD Parris Island is in the Lower Coastal Plain Physiographic Province. Elevations across the 8,047-acre depot range from sea level to 22-feet above mean sea level. Most of the Depot is within the 100-year flood plain. Drainage is typically to the nearest surface water body. Two primary aquifers are present within the area, the surficial aquifer and the Floridian Aquifer. These aquifers are separated by the Hawthorn/Cooper Marl Formation, which serves as a confining unit to the underlying Floridian Aquifer. The shallow aquifer generally consists of permeable fine- to medium-grained sands, with groundwater recharge to the water table through infiltration. The water table at MCRD Parris Island typically ranges between 0 and 10 feet below ground surface (bgs). Fluctuation, as a function of recharge, evaporation, and transpiration, has been observed to as much as 6.5 feet (Glowacz 1980). Shallow groundwater flow is generally toward the nearest surface water body. Due to its brackish nature, groundwater and surface water in the vicinity is not useable as a domestic water supply. Potable water in the area is supplied by the Beaufort-Jasper Water and Sewer Authority.

Site 35 is predominantly paved by asphalt and concrete, surrounded by a chain-link fence, and secured with a locked gate. Two single-story, concrete block buildings (Buildings 953 and 953C) support operations at the Site. Building 953, constructed in 1991, serves as a 90-day RCRA Hazardous Storage Facility for ignitable waste, miscellaneous hazardous waste, PCB-contaminated materials, and various other non-hazardous waste awaiting pickup for offsite disposal. Due to the active nature, this RCRA facility is not included in the scope of this study and will require further evaluation at time of closure. Building 953C, constructed as the natural resources warehouse in 2000, stores empty 55-gallon drums, deer stands, and various files.

Building 958, the Pollution Prevention Facility, adjacent to the west of the Site, has been used to provide temporary storage of textiles, cardboard, electronic, and fluorescent lights, prior to being shipped offsite for disposal/recycling since construction in 2007. Due to the recent construction date of Building 958, any spills would be handled under RCRA. Due to its recent construction date and operations, building 958 has not been included within the Site 35 boundary.

SITE LOCATION

1 inch = 2,000 feet

0 1,000 2,000 3,000 4,000 Feet

SOURCE: USGS 7.5 MINUTE PARRIS ISLAND (1980) AND BEAUFORT (1979) QUADRANGLES

FIGURE 10-1
SITE LOCATION MAP
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command

RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 5/2/2014
DRAWN BY: M. SENNE	TASK ORDER NUMBER: JM03

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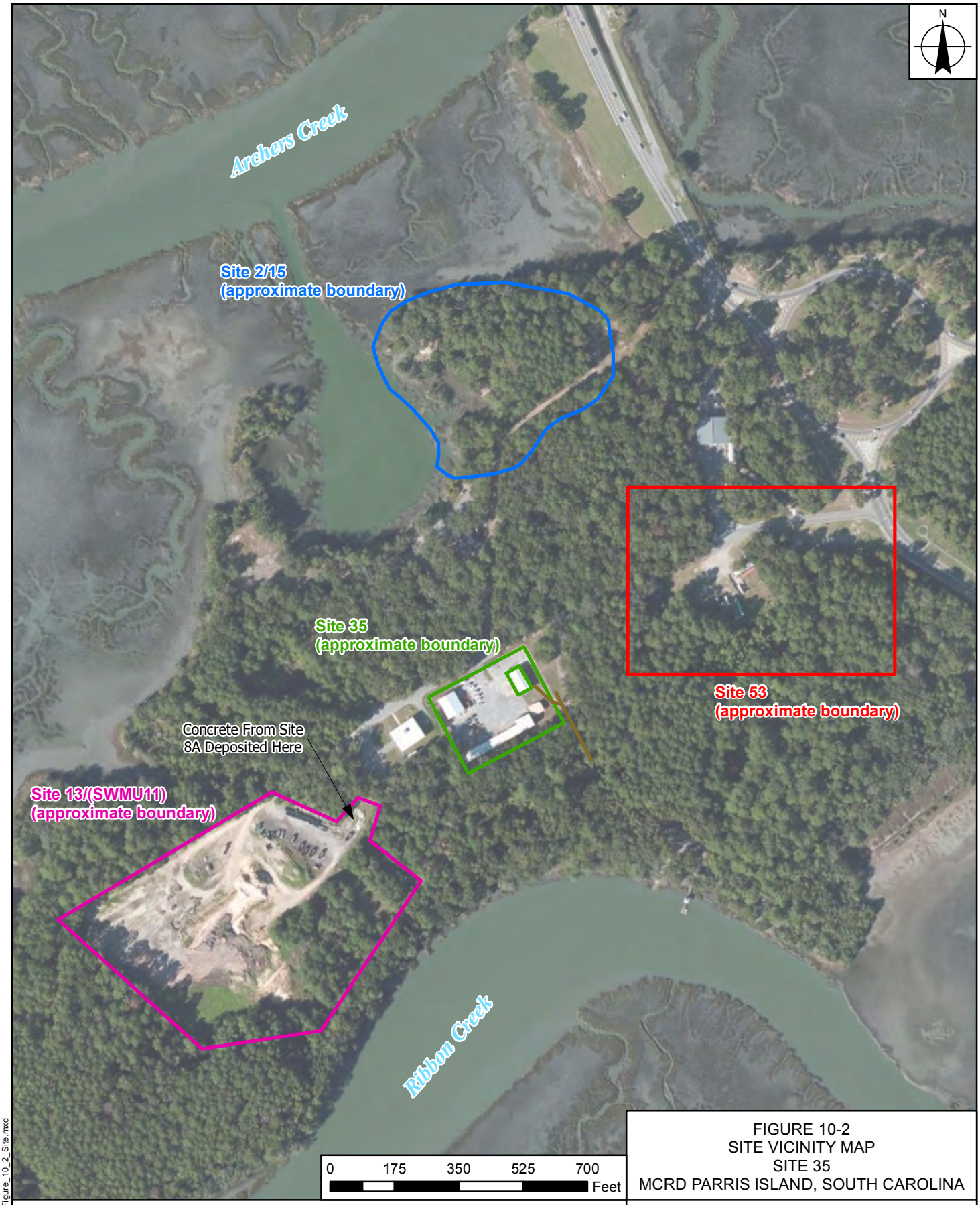

FIGURE 10-2
SITE VICINITY MAP
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

Legend

90-day RCRA Hazardous Storage Facility (Building 953)

NAVFAC Naval Facilities Engineering Command

RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 5/2/2014
DRAWN BY: M. SENNE	TASK ORDER NUMBER: JM03

SOURCE: GOOGLE EARTH PRO IMAGE DATE: JAN 28, 2011

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FIGURE 10-3
SITE MAP
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

Earthen Ditch (dry)

- Ditch Inferred from 2003 Sampling
- Ditch
- Gravel Road
- 90-day RCRA Hazardous Storage Facility (Building 953)
- 2004 Excavation

NAVFAC Naval Facilities Engineering Command

RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: M. SENNE	TASK ORDER NUMBER: JM03

SOURCE: GOOGLE EARTH PRO IMAGE DATE: JAN 28, 2011

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The Site's topography is generally flat, with a slight slope toward the south. Based on observations made during the site visit, drainage occurs via sheet flow over paved surfaces. Erosion was noted in the low-lying area immediately beyond the southern edge of the asphalt pavement, near the centerline of the Site. Sandy surface soils allow for rapid infiltration in limited unpaved areas of the Site. A single steel drainage grate was observed in the concrete surface in front of the support building in the eastern portion of the Site. Based on as-built drawings provided by MCRD Parris Island, the drain discharges to the earthen ditch at the southeast corner of the Site. The ditch extends offsite to the southeast, where it crosses beneath an unnamed dirt road and terminates in forested land. The ditch was dry at the time of the March 2012 site visit. No surface water features exist at the Site. Based on hydrogeological information obtained through previous studies (as summarized in Section 10.6), the shallow water table (surficial aquifer) is likely less than 10 feet bgs. Groundwater flow is anticipated to be toward Ribbon Creek, approximately 500 feet to the south. Between approximately 30 to 40 feet bgs, increasing clay content has been noted with depth. This clay layer is considered a localized semi-confining unit. As previously mentioned, recharge to the shallow aquifer occurs through infiltration, which is expected to be limited onsite due to the asphalt/concrete pavement.

The Site is bordered by undeveloped, forested land in all four directions. The nearest surface water features are Archers Creek, approximately 500 feet to the north and Ribbon Creek to the south. Two recreational facilities are located within 500 feet of the Site, including a picnic area at Archers Creek and a boat ramp and dock at Ribbon Creek. Both creeks drain to the west to Broad River, which empties to Port Royal Sound and the Atlantic Ocean.

10.4 Site and Vicinity Background

According to historical documents discussed in further detail in Section 10.5, Site 35 has operated as a DRMO salvage yard for materials generated at MCRD Parris Island, Marine Corps Air Station Beaufort, and Navy Hospital Beaufort, since 1964. From 1984 through 1985, the salvage yard also handled materials from Fort Stewart, Georgia. As previously discussed, recyclable materials, including paper, cardboard, wood, canvas, rubber, metals, spent ammunition casings, household appliances, electrical transformers, depleted fire extinguishers, and batteries are accepted and stored at the Site, pending offsite disposal. The Site also temporarily stores hazardous materials and wastes deemed potentially useful or salvageable, including expired shelf-life, damaged, and off-specification paints, glazing compounds, dry cleaning solvents, cleaning compounds, petroleum products, batteries, mercury amalgam, and recovered silver, pending resale or offsite disposal. No historical spills, releases, or incidents are documented at the Site.

In an effort to obtain additional information on the historical configuration of the Site and surrounding area, Resolution Consultants reviewed aerial photographs dated 1951, 1961, 1968, 1977, 1983, 1989, 1994, 1999, 2005, 2008, and 2011. Aerial imagery was obtained through Environmental Data Resources, Inc. and Google Earth. With the exception of a bridge crossing Archers Creek from Highway 802 to the north, Horse Island appears as undeveloped in the 1951 aerial photo. Site 35 was cleared by 1961, with additional clearing adjacent to the east and dirt roads apparent. Site 35 remains unpaved in the 1968 image. The existing causeway/bridge over Ribbon Creek, from MCRD Parris Island to the south, was constructed between 1968 and 1977. Portions of Site 35 appear to be paved by 1983. In 1994, a support building in the eastern portion and the storage shelter in the southern portion first appear at the Site. Similar to current conditions, two ASTs are apparent at the southeastern corner of the Site; however, they do not appear to be covered in the 1994 image. In 1999, a second support building appears at its current location in the northwestern corner of the Site. By 2005, Site 35 appears to be fully paved, with shelters erected over the storage area and ASTs. Between 2005 and 2008, a third building was constructed on formerly undeveloped land adjacent to the west. The 2008 Site configuration is similar to the present layout.

A teardrop-shaped surface water feature, to the south of Archers Creek, becomes evident between 1951 and 1961. From 1961 through 1994, much of the land around the southern perimeter of the teardrop appears cleared, with dirt roads providing access. In 1999 and 2005, additional forest growth is evident around the southern perimeter. Cleared areas are isolated to the southeastern, southern, and western edges of the teardrop feature. A second cleared area, approximately 600 feet to the west of Site 35 and to the south of Vieques Road, first appears on a 1968 image. From 1977 through 2005, this cleared area extends southward toward Ribbon Creek and eastward to within approximately 400-feet of Site 35. As further discussed in Section 10.6, various wastes and debris have been disposed in cleared areas at Horse Island since the mid-1960s.

10.5 Previous Studies

Information associated with previous studies at Site 35, as summarized herein, was obtained through the review of the following documents:

- *Interim Resource Conservation Act Facility Assessment Report* (A.T. Kearney, 1990)

- *Interim Remedial Actions for Sites SWMU 3, SWMU 35, SWMU 53, and SWMU 54* (Kemron 2005)

- *Site Inspection/Confirmatory Sampling (SI/CS) Report* (Tetra Tech NUS 2010)

- Informal memorandum regarding 2003 Sampling Efforts, as provided by U.S. EPA during the February 2013 partnering meeting

Solid Waste Management Unit (SWMU) 35 (Site 35) was identified during a 1990 Interim RCRA Facility Assessment (RFA), which consisted of a preliminary review of historical records and a visual inspection of the Site. During the visual inspection, cracked asphalt was observed beneath pallets of automotive batteries, therefore follow-up soil sampling was recommended. From 1995 through 2003, surface soil samples were collected from beneath cracked asphalt in the area used to store batteries, chemicals, and electrical transformers and from potential runoff areas, including the low-lying grassy area adjacent to the southern edge of the Site's asphalt cover and the earthen ditch, which exits the Site's southeast corner. For the purposes of this SAP, surface soil is defined as soil from 0 to 0.5 foot bgs. The majority of historical surface soil samples were analyzed for pesticides, PCBs, and select metals, while only two historical surface soil samples were tested for volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs). In 1999, one groundwater sample was collected to evaluate metals in shallow groundwater downgradient of the Site.

During 2005 drainage improvements, surface soil in the low-lying grassy area, at the southern edge of the Site, was excavated to a depth of 0.5 foot bgs. According to available information, the excavation footprint was 6 feet in width by 300 feet in length. An additional 3-foot-by-3-foot excavation was performed, to a depth of approximately 0.5 feet bgs, where the earthen ditch exits the Site at the southeast corner. Excavated soils were disposed offsite. No backfill occurred within the excavation footprint. At the direction of the Resident Officer in Charge of Construction, ten confirmatory soil samples, indicative of soil left-in-place, were collected from the base of excavated areas and analyzed for total pesticides, total PCBs, and TAL metals. Details associated with the historical excavation are documented in the *Final Completion Report for Interim Remedial Actions for Various Sites*, prepared by Kemron and dated 21 March 2005.

Historical sampling locations are depicted on Figure 10-4. Tables 10-1 through 10-3 compare results, indicative of soil left in place, to applicable screening levels, as defined in subsequent sections of this SAP. Results are depicted on Site maps, included as Figures 10-5 through 10-9, and discussed by analyte below.

VOCs

The VOCs carbon tetrachloride and tetrachloroethene were detected above U.S. EPA Protection of Groundwater Regional Screening Levels (RSLs) in surface soil beneath the storage area. Chloroform was detected above the Protection of Groundwater RSL and the U.S. EPA Ecological Screening Value (ESV) in surface soil beneath the storage area and also in surface soil where site drainage enters the earthen ditch at the southeast corner of the Site.

SVOCs

The SVOC, di-n-butyl phthalate, was reported above the Protection of Groundwater RSL in the surface soil sample collected at the entry point of the earthen ditch.

Pesticides

Only those pesticides measured above installation-specific background levels, as established during the Remedial Investigation (RI) for Site 3 (TetraTech NUS 1999), are discussed herein. Dieldrin was measured above the ESV in surface soil at the entry point of the earthen ditch. Concentrations of 4,4-DDE were measured above the Protection of Groundwater RSL and the ESV in surface soil samples collected from beneath the storage area and at the entry and midpoint of the earthen ditch. Concentrations of 4,4-DDT were measured above Residential RSLs, Protection of Groundwater RSLs, and the ESV in a surface soil sample collected near the storage area. Concentrations of 4,4-DDT also exceed the Protection of Groundwater RSL and the ESV in surface soil samples collected from the entry and midpoint of the earthen ditch.

PCBs

Two PCBs, Arochlor-1254 and -1260 were measured above Industrial, Residential, and Protection of Groundwater RSLs as well as ESVs in a surface soil sample collected near the storage area. Arochlor-1254 and 1260 were also measured above Residential RSLs, Protection of Groundwater RSLs, and ESVs at the entry point of the earthen ditch.

Historical Sampling Locations

- 1995 soil sampling location
- 1999 soil sampling location
- 2003 soil sampling location
- 2005 confirmatory soil sampling location
- 1999 groundwater sampling location

Earthen Ditch (dry)

- Ditch Inferred from 2003 Sampling
- Ditch
- Gravel Road
- 90-day RCRA Hazardous Storage Facility (Building 953)
- 2004 Excavation Area

Feet
0 50 100

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-4
HISTORICAL SAMPLING LOCATIONS
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: M. SENNE	TASK ORDER NUMBER: JM03

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Analyte	Industrial RSLs	Residential RSLs	Protection of Groundwater RSLs	ESVs
Carbon Tetrachloride	3000	610	0.15	1000000
Chloroform	1500	290	0.053	1
Tetrachloroethene	41000	8600	2.3	10

Notes:
all units ug/kg
RSLs = USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, Nov 2013
ESVs = Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998

1995 soil sampling location

Earthen Ditch (dry)

Ditch Inferred from 2003 Sampling

Ditch

Gravel Road

90-day RCRA Hazardous Storage Facility (Building 953)

2004 Excavated Area

Note: Results are for VOCs detected in µg/kg
J = Estimated Concentration
ND = Not Detected
BLUE text indicates exceedence of Protection of Groundwater RSLs
PINK text indicates exceedence of Protection of Groundwater RSLs and ESV

1 inch = 100 feet
0 25 50 75 100 Feet

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-5
HISTORICAL ANALYTICAL RESULTS
VOCs IN SURFACE SOIL
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: M. SENNE	TASK ORDER NUMBER: JM03

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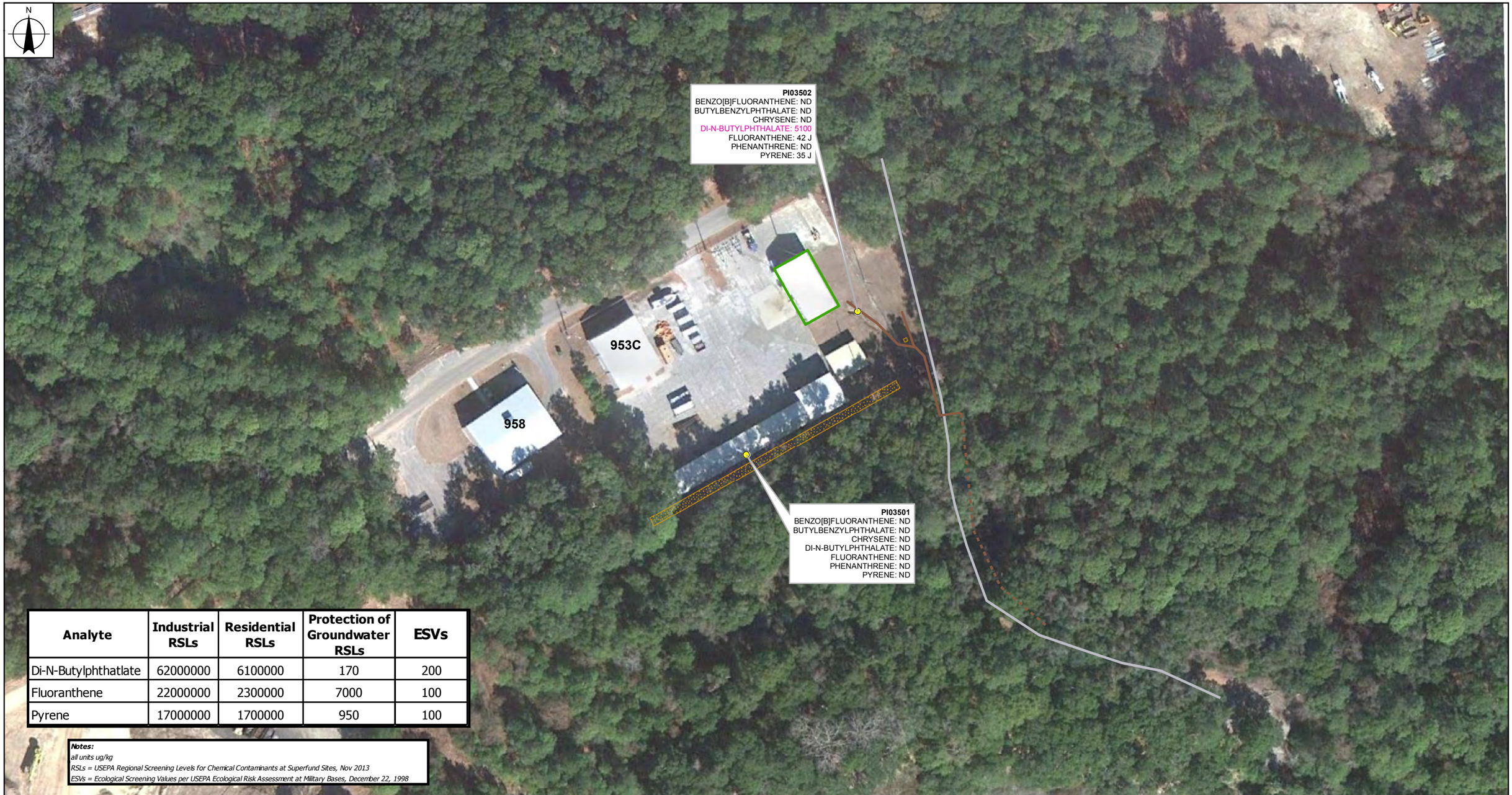

Analyte	Industrial RSLs	Residential RSLs	Protection of Groundwater RSLs	ESVs
Di-N-Butylphthatlate	62000000	6100000	170	200
Fluoranthene	22000000	2300000	7000	100
Pyrene	17000000	1700000	950	100

Notes:
all units ug/kg
RSLs = USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, Nov 2013
ESVs = Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998

1995 soil sampling location

Earthen Ditch (dry)

Ditch Inferred from 2003 Sampling

Ditch

Gravel Road

90-day RCRA Hazardous Storage Facility (Building 953)

2004 Excavated Area

Note: Results are for SVOCs detected in µg/kg
J = Estimated Concentration
ND = Not Detected
PINK text indicates exceedence of Protection of Groundwater RSLs and ESV

1 inch = 100 feet
0 25 50 75 100 Feet

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-6
HISTORICAL ANALYTICAL RESULTS
SVOCs IN SURFACE SOIL
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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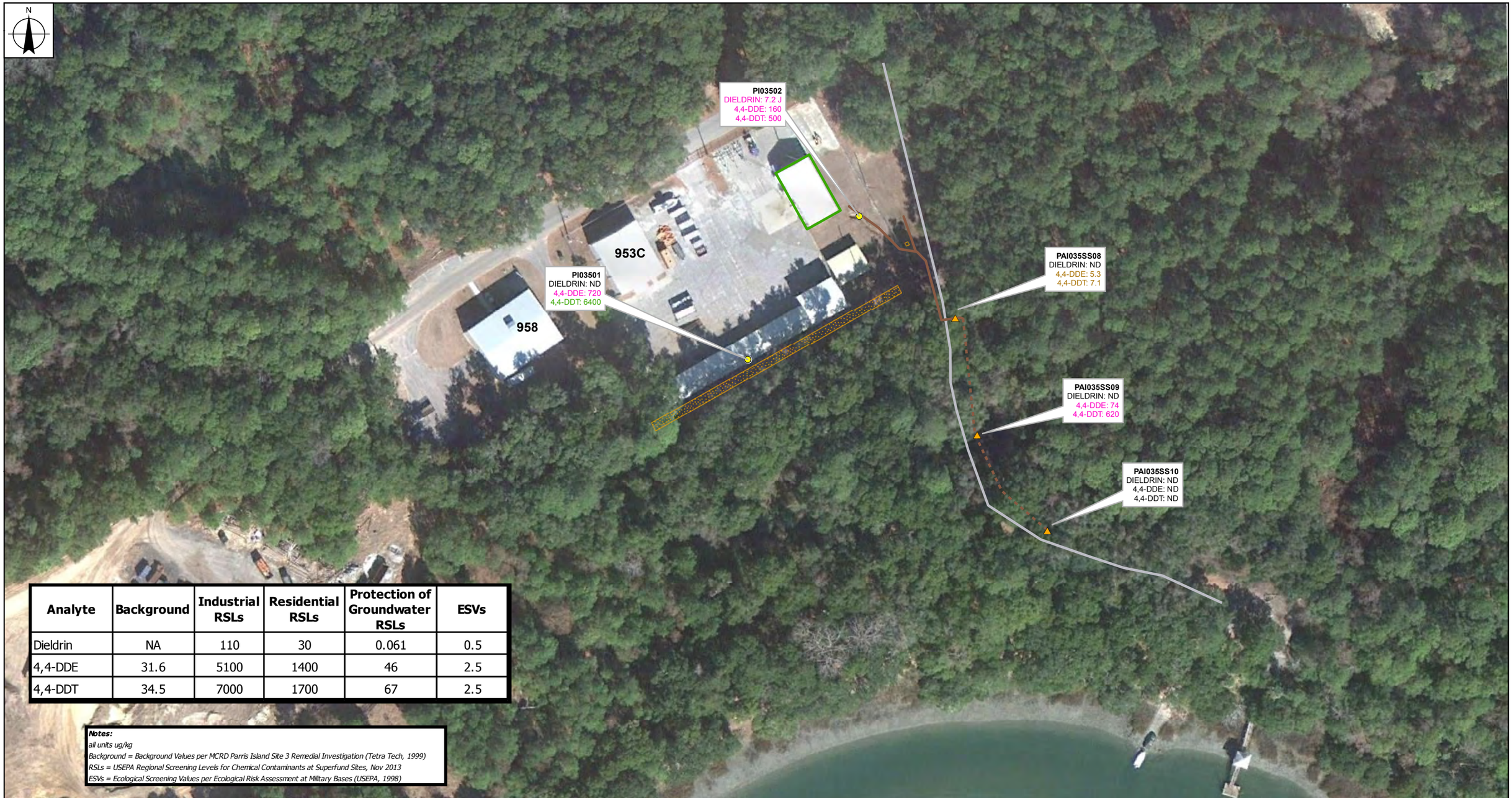

Analyte	Background	Industrial RSLs	Residential RSLs	Protection of Groundwater RSLs	ESVs
Dieldrin	NA	110	30	0.061	0.5
4,4-DDE	31.6	5100	1400	46	2.5
4,4-DDT	34.5	7000	1700	67	2.5

Notes:
all units ug/kg
Background = Background Values per MCRD Parris Island Site 3 Remedial Investigation (Tetra Tech, 1999)
RSLs = USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, Nov 2013
ESVs = Ecological Screening Values per Ecological Risk Assessment at Military Bases (USEPA, 1998)

1995 soil sampling location
2003 soil sampling location
90-day RCRA Hazardous Storage Facility (Building 953)
2004 Excavated Area

Earthen Ditch (dry)
Ditch Inferred from 2003 Sampling
Ditch
Gravel Road

Note: Results are for pesticides detected above background in µg/kg
ND = Not Detected
GREEN text indicates exceedence of Residential and Protection of Groundwater RSLs and ESV
PINK text indicates exceedence of Protection of Groundwater RSLs and ESV
BROWN text indicates exceedence of ESV

1 inch = 100 feet
0 25 50 75 100 Feet

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-7
HISTORICAL ANALYTICAL RESULTS
PESTICIDES IN SURFACE SOIL
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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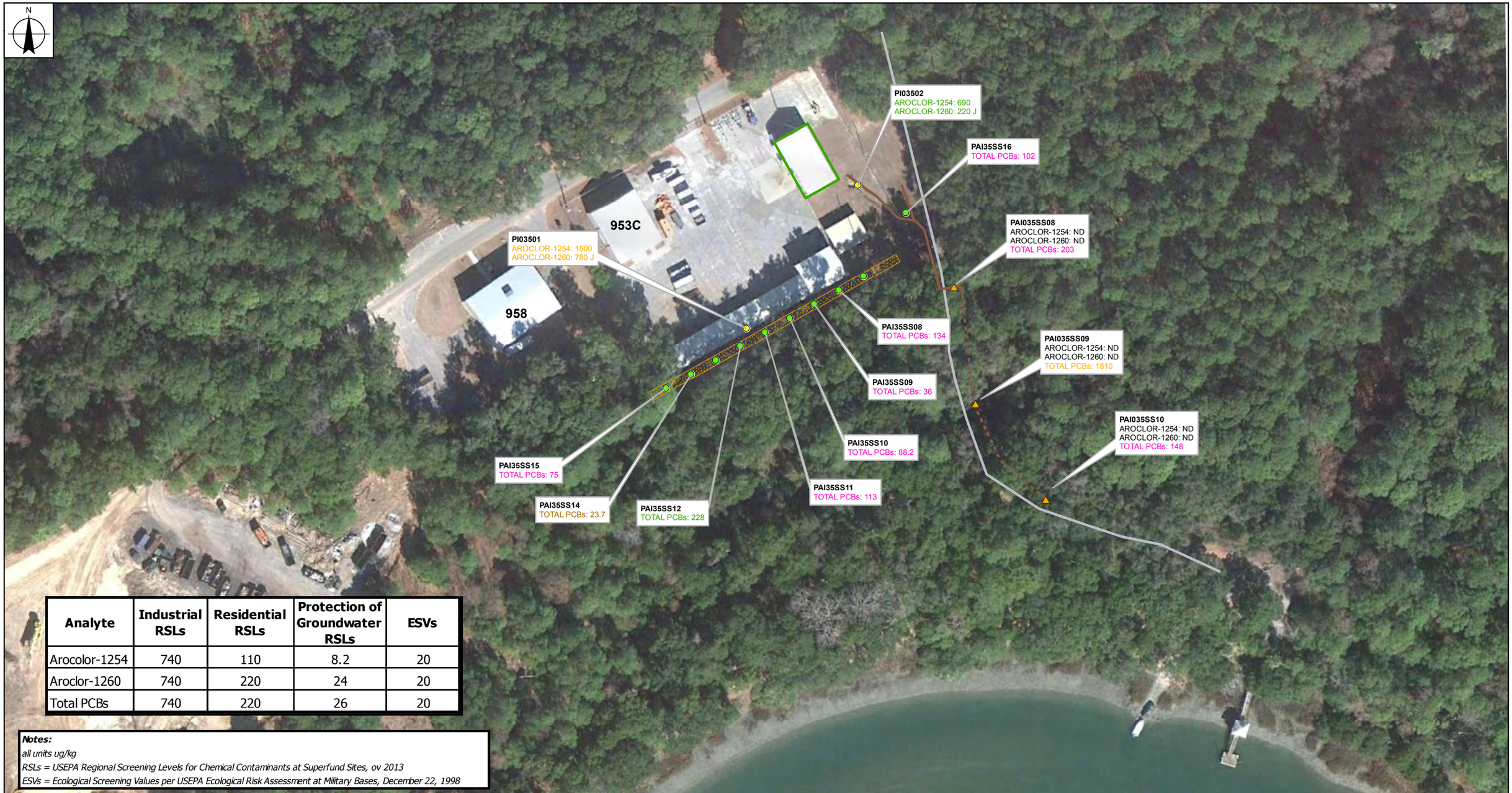

Analyte	Industrial RSLs	Residential RSLs	Protection of Groundwater RSLs	ESVs
Arocolor-1254	740	110	8.2	20
Aroclor-1260	740	220	24	20
Total PCBs	740	220	26	20

Notes:
all units ug/kg
RSLs = USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, ov 2013
ESVs = Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998

- 1995 soil sampling location
- 2003 soil sampling location
- 2005 confirmatory soil sampling location

Earthen Ditch (dry)

- Ditch Inferred from 2003 Sampling
- Ditch

- Gravel Road
- 90-day RCRA Hazardous Storage Facility (Building 953)
- 2004 Excavated Area

Note: Results are for PCBs detected in µg/kg
J = Estimated Concentration
ORANGE text indicates exceedence of Industrial, Residential, and Protection of Groundwater RSLs and ESV
GREEN text indicates exceedence of Residential and Protection of Groundwater RSLs and ESV
PINK text indicates exceedence of Protection of Groundwater RSLs and ESV
BROWN text indicates exceedence of ESV

1 inch = 100 feet
0 25 50 75 100 Feet

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-8
HISTORICAL ANALYTICAL RESULTS
PCBs IN SURFACE SOIL
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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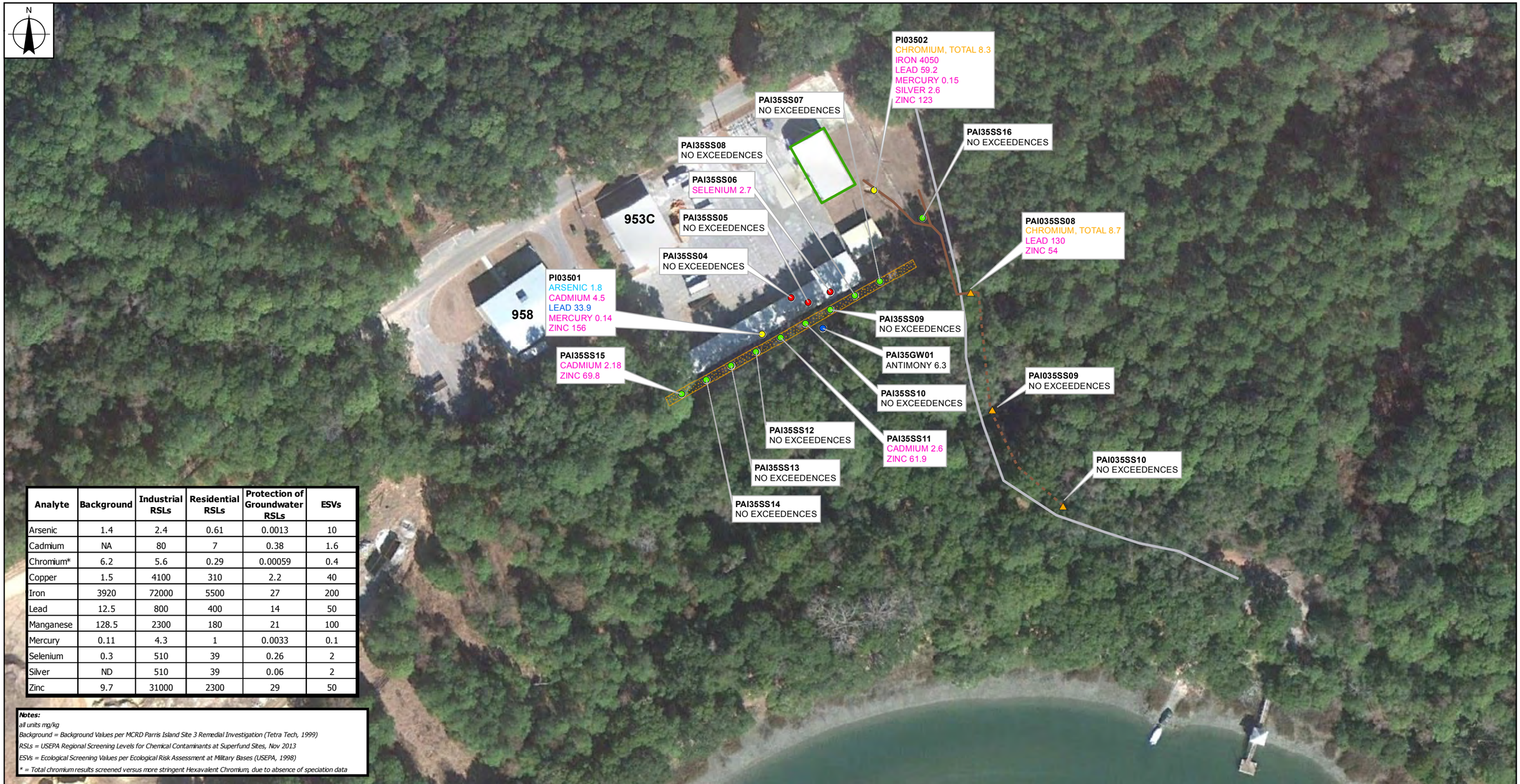

Analyte	Background	Industrial RSLs	Residential RSLs	Protection of Groundwater RSLs	ESVs
Arsenic	1.4	2.4	0.61	0.0013	10
Cadmium	NA	80	7	0.38	1.6
Chromium*	6.2	5.6	0.29	0.00059	0.4
Copper	1.5	4100	310	2.2	40
Iron	3920	72000	5500	27	200
Lead	12.5	800	400	14	50
Manganese	128.5	2300	180	21	100
Mercury	0.11	4.3	1	0.0033	0.1
Selenium	0.3	510	39	0.26	2
Silver	ND	510	39	0.06	2
Zinc	9.7	31000	2300	29	50

Notes:
all units mg/kg
Background = Background Values per MCRD Parris Island Site 3 Remedial Investigation (Tetra Tech, 1999)
RSLs = USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, Nov 2013
ESVs = Ecological Screening Values per Ecological Risk Assessment at Military Bases (USEPA, 1998)
** = Total chromium results screened versus more stringent Hexavalent Chromium, due to absence of speciation data*

- 1995 soil sampling location
- 1999 soil sampling location
- 2003 soil sampling location
- 2005 confirmatory soil sampling location
- 1999 groundwater sampling location

Earthen Ditch (dry)

- Ditch Inferred from 2003 Sampling
- Ditch
- Gravel Road
- 90-day RCRA Hazardous Storage Facility (Building 953)
- 2004 Excavated Area

Note: Soil results are for metals above background in mg/kg.
Groundwater results are for detected metals in µg/kg
PINK text indicates exceedence of Protection of Groundwater RSLs and ESV
BLUE text indicates exceedence of Protection of Groundwater RSLs
LIGHT BLUE text indicates exceedence of Residential and Protection of Groundwater RSLs
ORANGE text indicates exceedence of Industrial, Residential, and Protection of Groundwater RSLs and ESV

1 inch = 100 feet
0 25 50 75 100 Feet

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

FIGURE 10-9
HISTORICAL ANALYTICAL RESULTS
METALS DETECTED ABOVE BACKGROUND
VALUES IN SURFACE SOIL AND GROUNDWATER
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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Table 10-1
VOCs and SVOCs Detected in Soil (Left-In-Place)

					Sample Location:	**PI03501**	**PI03502**
					Area of Interest	storage	drainage
					Sample Date:	12/12/95	12/12/95
					Sample Depth (ft bgs):	0.5	0.5
Analyte	***EPA I-RSL (a)***	***EPA R-RSL (b)***	***EPA Protection of GW-RSL (c)***	***ESV (d)***	***Units***		
Volatile Organic Compounds							
Carbon Tetrachloride	3000	610	0.15	1000000	µg/kg	**3J c**	ND
Chloroform	1500	290	0.053	1	µg/kg	**3J cd**	**2J cd**
Tetrachloroethene	41000	8600	2.3	10	µg/kg	**3J c**	ND
Polynuclear Aromatic Hydrocarbons							
Di-N-Butylphthalate	62000000	6100000	170	200000	µg/kg	ND	**5100 c**
Fluoranthene	22000000	2300000	7000	100	µg/kg	ND	42J
Pyrene	17000000	1700000	950	100	µg/kg	ND	35J

Notes:

ND	=	Not detected
ft bgs	=	Feet below ground surface
EPA R-RSL (a)	=	US EPA Regional Screening Levels for Residential Soil, November 2013
EPA I-RSL (b)	=	US EPA Regional Screening Levels for Industrial Soil, November 2013
EPA Protection of Groundwater-RSL (c)	=	US EPA Soil Screening Level based on migration to groundwater and the Tap Water RSL, November 2013
ESV (d)	=	Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998
c	=	Exceeds EPA Protection of Groundwater-RSL
cd	=	Exceeds EPA Protection of Groundwater-RSL and ESV

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Table 10-2
Pesticides and PCBs Detected in Soil (Left-In-Place)

						Sample Location:	PI03501	PI03502	PAI03SS08	PAI03SS09	PAI03SS10	PAI35S08	PAI35S09	PAI35S10	PAI35S11	PAI35S12	PAI35S13	PAI35S14	PAI35S15	PAI35S16
						Area of Interest:	storage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage
						Sample Date:	12/12/95	12/12/95	12/18/03	12/18/03	12/18/03	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05
						Sample Depth (ft bgs):	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Analyte	*Background*	*EPA I-RSL (a)*	*EPA R-RSL (b)*	*EPA Protection of GW-RSL (c)*	*ESV (d)*	*Units*														
Pesticides																				
Dieldrin	--	110	30	0.061	0.5	µg/kg	ND	**7.2J cd**	ND	ND	ND	--	--	--	--	--	--	--	--	--
4,4-DDE	31.6	5100	1400	46	2.5	µg/kg	**720 cd**	**160 cd**	**5.3 d**	**74 cd**	ND	--	--	--	--	--	--	--	--	--
4,4-DDT	34.5	7000	1700	67	2.5	µg/kg	**6400 bcd**	**500 cd**	**7.1 d**	**620 cd**	ND	--	--	--	--	--	--	--	--	--
Polycyclic Biphenyls																				
Arocolor-1254	--	740	110	8.2	20	µg/kg	**1500 abcd**	**690 bcd**	ND	ND	ND	--	--	--	--	--	--	--	--	--
Aroclor-1260	--	740	220	24	20	µg/kg	**780J abcd**	**220J bcd**	ND	ND	ND	--	--	--	--	--	--	--	--	--
Total PCBs	--	740	220	26	20	µg/kg	--	--	**203 cd**	**1810 abcd**	**148 cd**	**134 cd**	**36 cd**	**88.2 cd**	**113 cd**	**228 bcd**	--	**23.7 d**	**75 cd**	**102 cd**

Notes:

ND	= Not detected
--	= Not available
ft bgs	= Feet below ground surface
Background	= Background Values per MMCRD Parris Island Site 3 Remedial Investigation (Tetra Tech, 1999)
EPA R-RSL (a)	= US EPA Regional Screening Levels for Residential Soil, November 2013
EPA I-RSL (b)	= US EPA Regional Screening Levels for Industrial Soil, November 2013
EPA Protection of Groundwater-RSL (c)	= US EPA Soil Screening Level based on migration to groundwater and the Tap Water RSL, November 2013
ESV (d)	= Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998
abcd	= Exceeds EPA I, R, and Protection of Groundwater-RSLs and ESV
bcd	= Exceeds EPA R and Protection of Groundwater-RSLs and ESV
cd	= Exceeds EPA Protection of Groundwater-RSL and ESV
d	= Exceeds ESV

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Table 10-3
Metals Detected in Soil (Left-In-Place)

						Sample Location:	PI03S01	PI03S02	PAI35SS04	PAI35SS05	PAI35SS06	PAI035SS08	PAI035SS09	PAI035SS10	PAI35S07	PAI35S08	PAI35S09	PAI35S10	PAI35S11	PAI35S12	PAI35S13	PAI35S14	PAI35S15	PAI35S16
						Area of Interest:	storage	drainage	storage	storage	storage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage	drainage
						Sample Date:	12/12/95	12/12/95	12/16/99	12/16/99	12/16/99	12/18/03	12/18/03	12/18/03	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05	12/03/05
						Sample Depth (ft bgs):	0.5	0.5	1	1	1	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Analyte	***Background***	***EPA I-RSL (a)***	***EPA R-RSL (b)***	***EPA Protection of GW-RSL (c)***	***ESV (d)***	***Units***																		
Metals																								
Arsenic	1.4	2.4	0.61	0.0013	10	mg/kg	**1.8 bc**	1.1	ND	ND	ND	ND	ND	ND	--	--	--	--	--	--	--	--	--	--
Cadmium	NA	80	7	0.38	1.6	mg/kg	**4.5 cd**	ND	ND	ND	ND	0.34	ND	ND	--	--	--	--	**2.6 cd**	--	--	--	**2.18 cd**	--
Chromium*	6.2	5.6	0.29	0.00059	0.4	mg/kg	5.9	**8.3 abcd**	3.4	3	2.8	**8.7 abcd**	1.4	0.76	4.78	3.45	3.09	3.36	3.74	3.5	3.11	3.25	2.69	--
Copper	1.5	4100	310	2.2	40	mg/kg	**22 c**	**13 c**	ND	ND	ND	**4 c**	1.1	ND	--	--	--	--	--	--	--	--	--	--
Iron	3920	72000	5500	27	200	mg/kg	2340	**4050 cd**	2150	2050	1730	1600	730	200	--	2190	2150	2270	2530	2570	2100	2180	1700	2120
Lead	12.5	800	400	14	50	mg/kg	**33.9 c**	**59.2 cd**	3.3	3.5	4.3	**130 cd**	10	3.5	--	--	--	--	--	--	--	--	--	--
Manganese	128.5	2300	180	21	100	mg/kg	94.8	66.6	23.1J	29.1J	23.7J	73	23	38	--	--	--	--	--	--	--	--	--	--
Mercury	0.11	4.3	1	0.0033	0.1	mg/kg	**0.14 cd**	**0.15 cd**	ND	0.08	0.06	ND	ND	ND	--	--	--	--	--	--	--	--	--	--
Selenium	0.3	510	39	0.26	0.81	mg/kg	ND	ND	ND	ND	**2.7 cd**	ND	ND	ND	--	--	--	--	--	--	--	--	--	--
Silver	NA	510	39	0.06	2	mg/kg	ND	**2.6 cd**	ND	ND	ND	ND	ND	ND	--	--	--	--	--	--	--	--	--	--
Zinc	9.7	31000	2300	29	50	mg/kg	**156 cd**	**123 cd**	10.8	5.8	3.8	**54 cd**	6.3	1.5	--	--	--	--	**61.9 cd**	--	--	--	**69.8 cd**	--

Notes:

ND	=	Not detected
--	=	Not available
ft bgs	=	Feet below ground surface
Background	=	Background Values per MMCRD Parris Island Site 3 Remedial Investigation (Tetra Tech, 1999)
EPA R-RSL (a)	=	US EPA Regional Screening Levels for Residential Soil, November 2013
EPA I-RSL (b)	=	US EPA Regional Screening Levels for Industrial Soil, November 2013
EPA Protection of Groundwater-RSL (c)	=	US EPA Soil Screening Level based on migration to groundwater and the Tap Water RSL, November 2013
ESV (d)	=	Ecological Screening Values per USEPA Ecological Risk Assessment at Military Bases, December 22, 1998
abcd	=	Above background (if available) and exceeds EPA I, R, and Protection of Groundwater-RSLs and ESV
bc	=	Above background (if available) and exceeds EPA R and Protection of Groundwater-RSLs
cd	=	Above background (if available) and exceeds EPA Protection of Groundwater-RSL and ESV
c	=	Above background (if available) and exceeds EPA Protection of Groundwater-RSL

** = Total chromium results screened versus more stringent Hexavalent Chromium, due to absence of speciation data*

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Concentrations of total PCBs were detected above Industrial, Residential, and Protection of Groundwater RSLs as well as ESVs in a surface soil sample collected at the midpoint of the earthen ditch. Total PCBs were also detected above Residential and Protection of Groundwater RSLs and ESVs in a surface soil sample collected from the base of the historical excavation completed in the low-lying grassy area to the south of the Site. Additionally, total PCBs were measured above Protection of Groundwater RSLs and ESVs in various surface soil sampling locations positioned at the base of the historical excavation and along the earthen ditch.

Metals

Eleven metals were detected in surface soil at concentrations exceeding background levels and at least one screening level. Total chromium concentrations exceeded background and Industrial, Residential, and Protection of Groundwater RSLs and the ESV for hexavalent chromium at the entry point and where the unlined ditch exits the Site. It should be noted that in the absence of speciation data, more stringent hexavalent chromium screening values were used in this overly conservative comparison. Arsenic exceeded the Residential RSL and the Protection of Groundwater RSL in the surface soil sample collected near the storage area; however, the lone arsenic exceedance is similar to its respective background level. Cadmium, copper, iron, lead, manganese, mercury, selenium, silver, and zinc were detected above background and Protection of Groundwater RSLs and ESVs; with the majority of the detections associated with the entry point and where the earthen ditch exits the property.

As presented on Table 10-4, antimony was the only metal detected above screening guidelines, consisting of the Maximum Contaminant Level (MCL) and the U.S. EPA RSL for Tap Water, in the lone groundwater sample collected at the storage area in 1999.

Results of historical studies at Site 35 were documented in the *SI/CS Report* (Tetra Tech NUS 2010), which concluded that given the Site's industrial setting, analytes detected in soil and groundwater do not pose significant threat to human health or the environment. The report recommended No Action/No Further Action. In a 23 April 2010 letter, SCDHEC commented that additional information is needed to concur with the No Further Action request. In a 23 April 2010 letter, the U.S. EPA disapproved the No Further Action request and recommended a RI/RFI at Site 35.

10.6 Additional Sites in the Vicinity

Due to uncertainty associated with historical disposal activities on Horse Island and their proximity to the Site, previous environmental investigations in the vicinity of Site 35 are discussed below and shown on Figure 10-2. Such information on potential off-site contamination sources/routes has been included in the CSM.

10.6.1 Sites 2/15 — Borrow Fill Pit and Dirt Roads

The 1990 RFA identified a landfill in the central portion of Horse Island, approximately 400-feet northeast of Site 35 and adjacent to the east of the Archers Creek teardrop feature, as SWMU 2 (Site 2) Borrow Fill Pit Landfill. According to the RFA, between 1918 to 1966, a mixture of lubricating oil, cutting oil, petroleum-based solvents, hydraulic fluids, and water-based coolants was applied to suppress dust along the road. The area in the vicinity of Site 2 was historically used as a source of fill dirt in the 1960s. According to the RFA, from 1966 through 1968, domestic trash, various debris, petroleum products, solid paint waste, paint strippers and thinners, cleaning rags, spent absorbent, aliphatic petroleum and chlorinated solvent sludge, chlorinated solvents, PCB contaminated oil, mercury amalgam, and beryllium wastes were disposed at Site 2. Reported quantities include receipt of 24,000 tons of domestic trash, 8,400-gallons of paint thinners, and 3,300-gallons of tetrachloroethene still bottoms. Available data at the time of the RFA indicated that groundwater at Site 2 was impacted by chloroform, 1,2-dichloroethane, chromium, and lead. Cadmium and chromium were detected in surface water, with chloroform detected in Site sediment. The RFA Report recommended an RI/RFI be performed at Site 2 and Phase II sampling be conducted at Site 15.

According to the RFI/RI Report submitted by Tetra Tech NUS in 2000, a combined investigation was performed at Site 2/15 between 1998 through 1999. Field activities included the installation of monitoring wells, and the collection of soil, sediment, groundwater, and surface water samples. Additionally, seventeen test pits were excavated at locations selected following a review of historical aerial photographs to identify areas where waste materials were potentially present beneath Site 2. The test pits were excavated to depths ranging from 2.5-feet and 8-feet bgs before termination upon reaching the shallow water table. Environmental samples collected from the base of select pits were analyzed for VOCs, acid and base neutrals, pesticides, PCBs, and metals.

Table 10-4
Metals Detected in Groundwater

			Sample Location:	**PAI35GW0101**
			Area of Interest:	storage
			Sample Date:	**12/14/99**
Analyte	***MCL (a)***	***Tap Water RSL (b)***	***Units***	
Metals				
Antimony	6	0.6	µg/L	**6.3 ab**
Barium	2000	290	µg/L	13.5
Calcium	NE	NE	µg/L	6260
Potassium	NE	NE	µg/L	924

Notes:

NE	= Not established
µg/L	= Micrograms per liter
MCL	= US EPA Maximum Contaminant Level, November 2013
Tap Water RSL	= US EPA Regional Screening Level for Tap Water, November 2013
ab	= Exceeds US EPA MCL and Tap Water RSL

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No evidence of historical waste was encountered during activities at the combined site. No metals were detected above background levels in surface or subsurface soil. Bis(2-ethylhexyl) phthalate and PCBs were each detected in one sediment sample, at a concentration above the ESV. Chloroform and arsenic were detected in groundwater samples; however, both concentrations were below MCLs. Bis(2-ethylhexyl) phthalate and arsenic were detected above human health risk based concentrations and silver was detected above the ESV in surface water. The risk assessment did not identify unacceptable risks and the RI/RFI Report recommended No Further Action, which was approved in the 2006 Record of Decision (U.S. EPA 2006).

10.6.2 Horse Island Disposal Areas

Site 13A, approximately 600 feet southwest of Site 35, consists of a 1,000-foot by 2,000-foot unlined landfill first permitted for the disposal of cellulosic wastes in 1976. According to the RFA Report, wood, yard waste, metal, and plastic have been known to be disposed of at Site 13A. There was no evidence of disposal or release of hazardous materials during the RFA. The RFA Report recommended Phase II sampling and suggested that activities at the landfill be closely monitored with respect to the depth of disposal trenches and the water table. Additionally, according to information provided by MCRD Parris Island, concrete debris, from restoration Sites 8A and 8B (the PCB Spill Area), was disposed in this area.

Site 53, the Horse Island Debris Area, approximately 250 to the east of the Site, historically served as an asphalt plant and disposal area for asphalt and concrete debris. According to information included in the most recent *Draft Final D2 MCRD Parris Island Federal Facilities Agreement Site Management Plan Amendment for Fiscal Year 2014*, trash removal and confirmatory sampling was performed at Site 53 in 2005. ESVs were exceeded in confirmatory soil samples and some debris remains. A preliminary assessment/site inspection is planned to further evaluate Site 53.

10.7 Conceptual Site Model

The initial CSM for Site 35, based on Resolution Consultant's review of historical records and reports and a preliminary site visit, is presented as Figure 10-10. Potential sources of contaminants, migration pathways, and receptors and exposure pathways are briefly discussed below.

10.7.1 Potential Sources of Contaminants

Potential onsite sources of contamination include historical releases from debris, scrap metal, appliances, equipment, transformers, chemicals, and petroleum products stored at the Site. Nature and extent data to date can be summarized as follows:

- VOCs, SVOCs, pesticides, PCBs, and metals have been detected above screening levels in historical surface soil samples collected at the Site, with highest concentrations noted in storage and drainage areas.

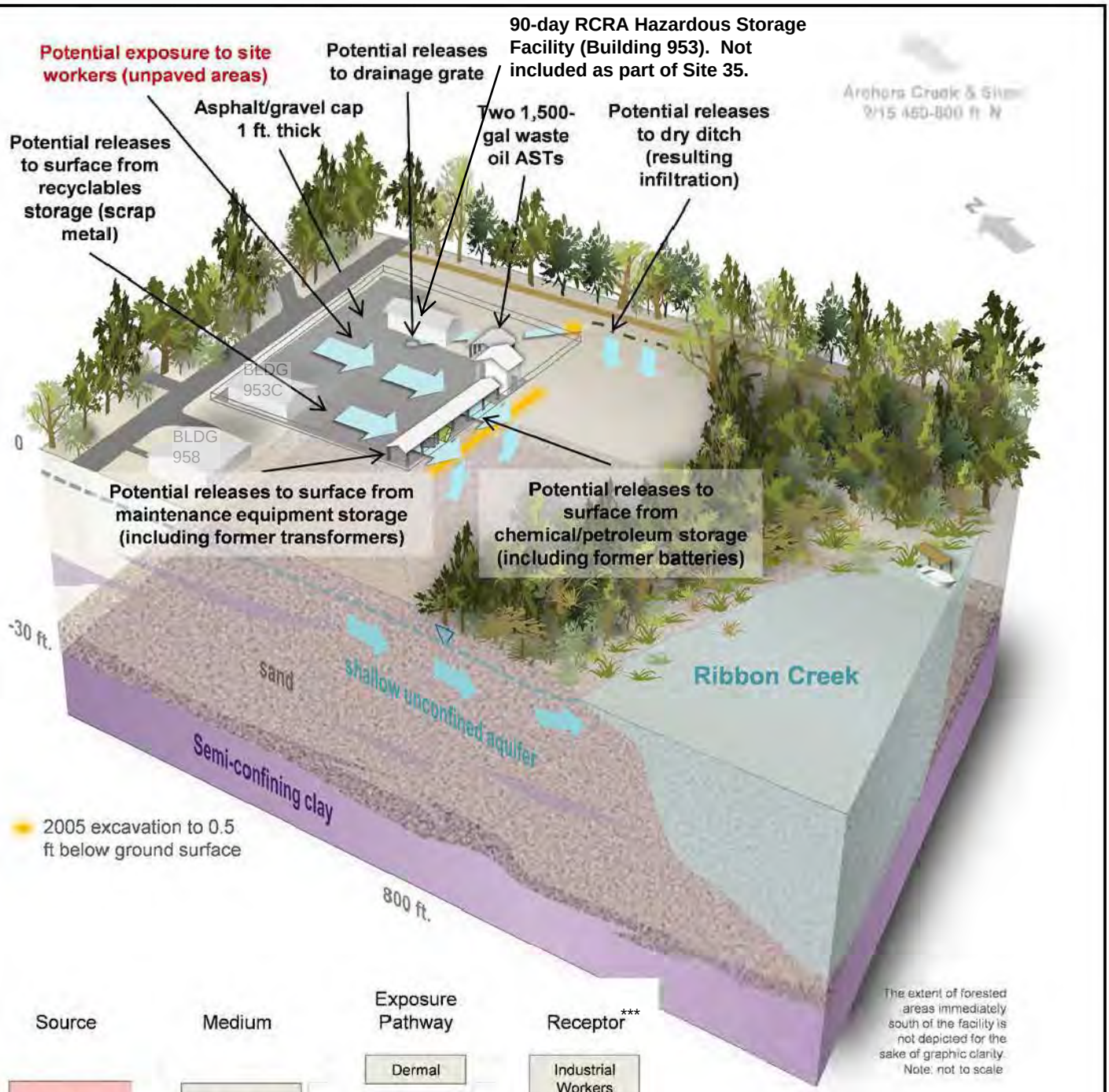
- No subsurface soil samples have been collected to date.

- Antimony was detected slightly above the MCL in Site groundwater, which to date, has only been tested for TAL metals, cyanide, and tin.

Due to the limitations of the current dataset, impacts to environmental media resulting from historical operations at the Site cannot be fully evaluated at this time. Therefore, an RI/RFI is warranted to assess conditions at Site 35.

10.7.2 Contaminant Migration Pathways

The Site, with the exception of a relatively small, maintained grassy area at the eastern boundary, has been paved by asphalt or concrete since at least 1990 (A.T. Kearney 1990). In general, any potential releases since that time would have drained over the paved surface to the low-lying area along the Site's southern boundary. Surface soil in this drainage area was excavated to a depth of 6 inches; however, confirmatory samples collected from the base of the excavation indicate that impacted soil remains in place. Potential releases within the Site's paved storage area may have followed localized cracks to the underlying gravel and sandy soil. Furthermore, any potential historical releases, occurring prior to paving, may have impacted soil beneath the existing surface cover. A steel grate currently collects drainage near the front of the easternmost support building. This grate presumably drains to the earthen ditch at the southeastern corner of the Site. The ditch is comprised of dry, sandy soil. Upon reaching unpaved terrain, including the earthen ditch, potential contamination could infiltrate to subsurface soil and possibly to shallow groundwater. The water table is likely less than 10 feet bgs at the Site, with flow toward Ribbon Creek to the south.



Source	Medium	Exposure Pathway	Receptor***
Onsite Release	Surface Soil	Dermal Ingestion Inhalation	Industrial Workers Ecological Receptors
	Subsurface Soil	Dermal Ingestion Inhalation	Industrial Workers Ecological Receptors
Offsite Release from Upgradient Site	Groundwater	Dermal Ingestion Inhalation	Industrial Workers Ecological Receptors

*** = Although not currently present or anticipated via future landuse, hypothetical future residents will be evaluated to provide an indication of potential risk if MCRD Parris Island were to close and be redeveloped for residential use.

Note: Figure depicts potentially complete receptors and exposure pathways. Results of this study will be used to refine this Conceptual Site Model.

FIGURE 10-10
CONCEPTUAL SITE MODEL
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 11/29/2012
DRAWN BY: L. HUGHES	TASK ORDER NUMBER: JM03

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10.7.3 Receptors and Exposure Pathways

Site 35, situated on Horse Island, is approximately 1 mile to the northwest of Parris Island and 1.5 miles south of the nearest residences at Port Royal. There are no residences on Horse Island. The Depot has a single point of access for vehicular traffic, with military police stationed at the entrance to limit outside visitors. Future land use at the Site is expected to remain industrial, consistent with the current use. Groundwater is not used as a potable water supply at the Site or in the vicinity of the Site, nor is it expected to be in the future. Only maintenance and industrial workers, who deliver and sort recyclables and accept stored materials, are allowed entry to Site 35. Since the Site is predominantly paved, use by terrestrial receptors is negligible. However, ecological receptors could potentially come into contact with impacted soil in the low-lying area to the south of the fenced-in storage area and in the dry, earthen ditch.

The Site is bordered by temperate evergreen forest, consisting primarily of oaks, pines, magnolia, red bay, sweet bay, yaupon holly, and saw palmetto, in all four directions. The understory is comprised of various ivy and shrubs. Although this forested area is not particularly sensitive, numerous wildlife species utilize this habitat. Estuarine areas, associated with Archers Creek to the north and Ribbon Creek to the south, provide a habitat for numerous fish, crustaceans, and other aquatic species. One Federally-listed endangered species, the wood stork, could reside in or along surface waters in the vicinity.

The Bald Eagle has been delisted by the United States Fish and Wildlife Service; however, it is still protected under the Bald and Golden Eagle Protection Act. Both Archers Creek and Ribbon Creek support commercial and recreational fishing, shellfish harvesting, boating, and water recreation.

Potential transport mechanisms to be considered include direct contact, air transport, bio-uptake, and leaching to groundwater. Potential exposure routes include ingestion, dermal contact, inhalation, ingestion of plants/animals, and inhalation of particulates. Potentially complete pathways and exposure routes include:

- Ingestion and dermal contact with surface soil and subsurface soil by onsite workers.
- Inhalation of particulates from surface soil and subsurface soil by onsite workers.
- Dermal contact with, ingestion of, and inhalation of chemicals leached to groundwater by onsite workers.

- Dermal contact with and ingestion of surface soil and subsurface soil and shallow groundwater by ecological receptors.

Although future residents are not receptors under current or expected future land use, they will be evaluated to provide an indication of potential risks if MCRD Parris Island were to close and be redeveloped for residential use.

SAP WORKSHEET #11: PROJECT QUALITY OBJECTIVES/SYSTEMATIC PLANNING PROCESS STATEMENTS

(UFP-QAPP Manual Section 2.6.1)

11.1 Overview

The U.S. EPA's *Guidance for Systematic Planning Using the DQO Process* (U.S. EPA, 2006) describes the process used to determine the type, quantity, and quality of data necessary to support decision-making regarding current site conditions and future site management. Inherent in the development of DQOs is a systematic and logical approach intended to yield an efficient sampling design based on accepted levels of potential decision errors. The following sections present the problem statement, decision criteria, information inputs, and the analytical approach, along with decision rules to be used during the execution of the RI/RFI at Site 35. Performance criteria, to assure data used to make project decisions are of sufficient quality, are also presented herein.

11.2 Problem Statement

Results generated during previous investigations, indicate that surface soil and groundwater may have been impacted by historical operations; however, the current dataset is insufficient to support a decision concerning whether or not future action is required.

11.3 Goals of the Study

The RI/RFI will investigate potential contamination and assess potential threats to human health and the environment at the Site, through screening comparisons only. To fill data gaps, soil and groundwater will be evaluated for COPCs, including VOCs, SVOCs, pesticides, PCBs, and TAL metals, as defined based on historical detections and operations at the Site. Findings will provide information to support the development, evaluation, and selection of appropriate response alternatives, if necessary.

Principal study questions (PSQs), developed to define decision statements to resolve the problem are as follows:

PSQ1: Do concentrations in surface and subsurface soils exceed applicable screening levels/background concentrations, thus requiring further investigation and/or risk evaluation?

PSQ2: Do concentrations in shallow groundwater exceed applicable screening levels, thus requiring further investigation and/or risk evaluation?

11.4 Information Inputs

The inputs needed to resolve the project problem identified in Section 11.3 include field observations and measurements, chemical data, site feature locations, and screening levels as described below. Observations made during Site visits, historical information obtained through review of records and aerial photographs, and data collected during previous studies, as summarized in Worksheet #10, were used to develop the CSM and sampling plan. Specific informational inputs, to serve as the basis for decisions during the execution of this project, include the following:

- Chemical Data: Soil and groundwater chemical data will be collected to determine if target analytes are present and assess potential threats to human health and the environmental at the Site. Concentrations of any detected target analytes will be compared to their respective screening levels, to support the decision making process.

- Geologic/Hydrogeologic Information: Information on site-specific geology will be obtained through observations made during soil boring activities. Onsite geologists/engineers will use the Unified Soil Classification System to thoroughly describe soil characteristics. Depth to groundwater measurements will be obtained at each well to provide information on site-specific hydrogeology and groundwater flow direction. Such information will be incorporated into the CSM.

- Field Screening: Results of field screening, including the use of a photoionization detector (PID) to measure organic vapors, will assist with characterization and in selecting samples to be submitted for laboratory analysis. Results of field screening are not considered definitive; thus, any decision making will be made using chemical data.

- Field investigation parameters: Parameters, including pH, temperature, specific conductivity, oxidation reduction potential, dissolved oxygen, and turbidity, will be used to confirm groundwater samples are representative of the formation being investigated. Field parameters will also be used to assist with understanding of the nature of contamination.

- Screening Levels: Chemical data will be compared to screening levels to determine COPCs at the Site. Applicable screening levels for this study are presented by media and potentially impacted receptor below.

  Human Health- Human health screening levels will consist of U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites (RSL) as documented at the following website:

  http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm

  Human health screening will include soil and groundwater data. Soil data will be compared to residential and protection of groundwater RSLs. Groundwater data will be compared to MCLs and tap water RSLs for such analyte. Analytes measured at concentrations exceeding human health screening levels and background concentrations will be retained as COPCs. In the event that COPCs are determined to exist at the Site, further investigation/risk evaluation may be required. Note that the most recent version of the RSL tables will be used for data evaluation following fieldwork.

  Ecological- Ecological risk screening for soil and groundwater data will be facilitated using corresponding ecological screening values (ESVs). Surface water ESVs will be used to screen groundwater data since ecological receptors may become exposed to groundwater upon discharge to surface water. U.S. EPA Region 4 ESVs for soil and surface water will be used for ecological risk screening, as provided at the following websites:

  http://www.epa.gov/region4/superfund/images/allprogrammedia/pdfs/tsstablesoilvalues.pdf

  http://www.epa.gov/region4/superfund/programs/riskassess/ecolbul.html

  If a constituent is not listed in the Region 4 ESVs, alternative screening values will be used. Alternative ESVs to be considered may include U.S. EPA Ecological Soil Screening Levels and/or Screening Quick Reference Tables as summarized by the National Oceanic and Atmospheric Administration. When screening values other than ESVs are cited, they will be clearly identified and their acceptability and approval for their use will be coordinated with the Navy, MCRD Parris Island, SCDHEC, and U.S. EPA before incorporating these values into the ecological screening process. Analytes measured at concentrations exceeding these ecological

screening levels and background concentrations will be retained as COPCs. In the event that COPCs are determined to exist at the Site, further investigation/risk evaluation may be required.

- Project Action Levels: A comprehensive summary table of all screening levels and/or criterion that may be used during this study is included as Appendix C. Worksheet #15 summarizes the lowest project action level (PAL) currently identified based on the applicable screening levels, as defined above. The laboratory selected for current work and any laboratories selected for future work are expected to achieve limits of quantitation (LOQs) that are low enough to measure constituent concentrations less than the Worksheet #15 PAL. Appendix C and Worksheet #15 provide sources for screening levels considered in PAL development; however, note that the most recent version of screening levels will be considered during the data evaluation process.

  Analytical data reported by the laboratory will use the following reporting conventions:

  - All concentrations less than the limit of detection (LOD) will be considered non-detects and will be reported as the LOD value with a "U" qualifier

  - Concentrations between the LOD and LOQ will be reported as estimated values with a "J" qualifier

  - In the event that a target analyte has a Worksheet #15 PAL between the LOD and LOQ, the "J" flagged data will be accepted to achieve project goals

  - The inability to quantify select chemicals to PAL levels with confidence will be addressed in the RI/RFI Report. The MCRD Parris Island Partnering Team will be involved in rendering the final conclusion regarding adequacy of the data.

- Background Concentrations: Background concentrations are generally considered to be the level or amount of a constituent found in common areas not associated with a particular contaminant release. If concentrations of constituents in soil samples from the site exceed background values, then the site may have residual contamination. Comparison to background concentrations of constituents also provides a relative idea of the degree of contamination. Such comparisons are helpful when addressing contamination in areas that may have been influenced by numerous anthropogenic (nearby roads) and natural activities

(weathering, flooding, wind dispersion). To help address the principal study questions, the concentrations of pesticides and metals (greatest and average) detected in soil will be compared to background concentrations. It should be noted that background soil values as documented in the approved RFI/RI Report for Site 3 Causeway Landfill (Tetra Tech NUS 1999) were used during the initial comparison of historical results, as presented in Worksheet #10. Resolution Consultants understands that the screening protocol for the existing Marine Corps Air Station Beaufort background dataset from the *RCRA Facility Investigation Report for SWMU 3* (Tetra Tech, 2006), commonly used for comparisons at other MCRD Parris Island CERCLA sites, is currently being re-evaluated by the MCRD Parris Island Partnering Team. Pending results of discussions by the MCRD Parris Island Partnering Team and prior to screening, background values to be used during this study will be specified and the proposed method of screening will be submitted for approval by the Navy, MCRD Parris Island, SCDHEC, and U.S. EPA.

- Regulatory Correspondence/Planning Meetings: Sampling network discussions and decisions made in stakeholder planning meetings, as presented in Worksheet #9, will be recorded and considered during project execution.

11.5 Study Area Boundaries

Site 35 is adjacent to the south of Vieques Road, on unpopulated Horse Island. At approximately 1.5-acres, Site 35 is predominantly paved by asphalt and concrete, surrounded by a chain-link fence, and secured with a locked gate. Three single-story, concrete block buildings support the site's current storage operations. Based on historical information on site use, including aerial photographs, the horizontal boundary is defined as the outer perimeter of the fenced storage area, plus the low-lying grassy area to the south and the earthen drainage ditch to the southeast. The vertical boundary for soil is assumed to be the soil/water table interface, estimated at less than 10 feet below ground surface. Data will be collected to evaluate potential releases to both surface and subsurface soil. The vertical boundary for groundwater is assumed to be the shallow (water table) aquifer, estimated to be encountered at less than 10 feet below ground surface. Groundwater samples will be collected from monitoring wells installed in the shallow formation. The temporal boundaries of the SI include the estimated duration of field activities.

11.6 Analytic Approach

The RI/RFI will investigate potential contamination and assess potential threats to human health and the environment, through screening comparisons. The sampling design has been chosen to represent

a worst-case estimate of constituents. Biased sampling locations will be collected based on historical information, observations made during the March 2012 Resolution Consultants site visit, and input from the DQO planning team. Sampling locations may be adjusted based on field observations and professional judgment.

Initial determinations on the need for follow-up action will be based on whether analytical data exceed applicable screening levels and background concentrations. Good faith efforts were undertaken to ensure that contaminants can be measured at the lowest achievable levels for the selected analytical method. However, as summarized in Worksheet #15, in some cases, current technology used by the analytical methods cannot achieve respective PALs. Any inability to quantify select chemicals to PALs with confidence will be addressed in the RI/RFI Report, with the MCRD Parris Island Partnering Team involved in rendering the final conclusion regarding adequacy of the data. Prior to implementing decision rules, the TOM, project chemist, and risk managers will review the analytical data, ecological screening results, and effective ecological pathways, as well as human health screening results and effective human health pathways, to determine if the site poses any unacceptable risk. If required by an ecological exceedance, ecological receptors foraging areas will be estimated to prepare a basic hazard index in accordance with Navy Ecological Risk Policy (STEP 3-C). The resulting decision rules are summarized below.

Decision Rule 1: If concentrations in surface and subsurface soils contain constituents at concentrations below human health screening levels/background concentrations, then the constituent will not be retained as a chemical of potential concern for further investigation and/or risk evaluation. If concentrations in surface and subsurface soils contain constituents at concentrations above human health screening levels/background concentrations, then the constituent will be retained as a chemical of potential concern for further investigation and/or risk evaluation.

Decision Rule 2: If concentrations in surface and subsurface soils contain constituents at concentrations below ecological screening levels/background considerations, then the constituent will not be retained as a chemical of potential concern for further investigation and/or risk evaluation. If concentrations in surface and subsurface soils contain constituents at concentrations above ecological screening levels/background considerations, then the constituent will be retained as a chemical of potential concern for further investigation and/or risk evaluation.

Decision Rule 3: If groundwater contains constituents at concentrations below human health screening levels, then the constituent will not be retained as a chemical of potential concern for further investigation and/or risk evaluation. If groundwater contains constituents at concentrations above human health screening levels, then the constituent will be retained as a chemical of potential concern for further investigation and/or risk evaluation.

Decision Rule 4: If groundwater contains constituents at concentrations below ecological screening levels, then the constituent will not be retained as a chemical of potential concern for further investigation and/or risk evaluation. If groundwater contains constituents at concentrations above ecological screening levels, then the constituent will be retained as a chemical of potential concern for further investigation and/or risk evaluation.

Decision Rule 5: If elevated levels of contaminants are fully delineated to applicable screening levels for soil and groundwater, then no additional data collection will be required. If elevated levels of contaminants are not fully delineated to applicable screening levels for soil and groundwater, then the MCRD Parris Island Partnering Team will review the data and determine if additional data should be collected.

11.7 Performance or Acceptance Criteria

Sampling design errors are a function of the selection of sample locations or analytical methods used to characterize the Site. In this case, a judgmental approach was used in developing the sampling design. Initial sampling locations are biased to areas most likely to be contaminated. Decision errors are reduced by subjective definition of the factual basis for the judgment. Decision uncertainty is managed by increasing the density of sampling locations in areas where there is high uncertainty in the correctness of a decision. Every effort has been made to minimize decision errors/uncertainty through obtaining comprehensive historical information with regards to Site 35 and incorporating such information into the CSM, prior to field deployment.

Worksheet #15 provides the analyte list, laboratory detection limits, and PALs that will be used to answer PSQs associated with this RI/RFI. Accuracy and precision of laboratory measurements will be based on DoD *Quality Systems Manual, version 4.2* (DoD 2010). Validated laboratory results will be used for characterization and overall decision making. Analytical methods, presented on Worksheet #19, were chosen that would achieve the best detection limits to answer the PSQs. Resolution Consultants will subcontract a South Carolina certified laboratory to perform analysis.

All data will be validated by the Project Chemist (or designee) against the analytical methodology requirements and measurement performance criteria presented in this SAP (Worksheets #12, 28, 34, 35, and 36), with findings documented in a data quality assessment report. Measurement error is further managed through the use of SOPs and data quality management. Copies of Laboratory SOPs are included in Appendix B.

SAP WORKSHEET #12: FIELD QUALITY CONTROL SAMPLES

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table — Field QC Samples				
QC Sample	**Analytical Group**	**Frequency**	**Data quality Indicators**	**Measurement Performance Criteria**
Equipment Rinsate Blank	All analytical groups	One per 20 samples per matrix per sampling equipment	Bias/Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.
Trip Blanks	VOCs	One per cooler containing VOC samples	Bias/Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.
Field Duplicate	All analytical groups	One per 10 field samples	Precision	Values > 5X LOQ: RPD must be ≤30 (aqueous), ≤50 (solids)
Matrix Spike/Matrix Spike Duplicate	All analytical groups	One pair per 20 field samples	Accuracy/Bias/ Precision	Percent recoveries — DoD QSM Limits RPD must be ≤ 30 (organics) RPD must be ≤ 20 (metals)
Cooler Temperature Indicator	All analytical groups	One per cooler	Representativeness	Temperature must be above freezing and less than or equal to 6 °C.

Notes:

QC = Quality control
VOCs = Volatile organic compounds
LOQ = Limit of Quantitation
RPD = Relative Percent Difference
DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010 or the most recent version at the time of sampling.
°C = Degrees Celsius

SAP WORKSHEET #13: SOURCES OF SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Historical Background Information, Data	*Interim Resource Conservation Act Facility Assessment of United States Marine Corp (USMC) Recruit Depot, Parris Island, South Carolina, EPA I.D. No. SC6170022762,* 1990	A.T. Kearney	Background information was used in planning of the sampling effort.	None
Historical Background Information, Data	*Interim Remedial Actions for Various Sites — SWMU 3, SWMU-35, SWMU-53, SWMU-54*, 2005	Kemron	Background information was used in planning of the sampling effort.	None
Historical Background Information, Data	*Site Inspection/ Confirmatory Sampling Report for Site/SWMU 4, Site/SWMU 5, Site/SWMU 7, Site 9/SWMU8, Site 13C/SWMU13, Site/SWMU 16, SWMU 27, and SWMU 35, Marine Corps Recruit Depot, Parris Island, South Carolina*, January 2010	Tetra Tech NUS	Background information was used in planning of the sampling effort.	None

SAP WORKSHEET #14: SUMMARY OF PROJECT TASKS

(UFP-QAPP Manual Section 2.8.1)

Field activities will be performed in accordance with applicable state and federal guidance (when available), including SCDHEC Well Standards R.61-71 and U.S. EPA Region 4 Science and Ecosystem Support Division Field Branches Quality System and Technical Procedures found at the following web site: http://www.epa.gov/region4/sesd/fbqstp/. Applicable U.S. EPA and SCDHEC technical procedures are identified in Worksheet #21 and included as Appendix A. Planned field activities are as follows:

- Mobilization/Demobilization
- Utility Clearance
- Site-Specific Health and Safety Training
- Equipment Calibration
- Soil Sampling
- Monitoring Well Installation and Development
- Groundwater Level Measurements
- Groundwater Sampling
- Global Positioning System (GPS) Locating
- Land Surveying
- Waste Handling
- Decontamination
- Site Restoration
- Field Documentation

Additional project-related tasks include:

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

Mobilization/Demobilization

Mobilization will consist of the delivery, assembly (in satisfactory working order), and secure storage of necessary equipment, materials, and supplies, along with the acquisition of personnel and vehicle base access badges. The Resolution Consultants FOL or designee will

coordinate with MCRD Parris Island POC to identify appropriate locations for the temporary storage of equipment and supplies.

Site-specific health and safety training for all Resolution Consultants field personnel and subcontractors will be conducted as part of mobilization. There are no specialized/non-routine project-specific training requirements or certifications needed by personnel to successfully complete the project or tasks. All field personnel will have appropriate training to conduct the field activities to which they are assigned. Each site worker will be required to have completed the Occupational Safety and Health Administration 40-hour Hazardous Waste Operations and Emergency Response course and 8-hour refresher, if applicable. Additional health and safety requirements are addressed in greater detail in the site-specific HASP.

Demobilization will consist of the prompt and timely removal of equipment, materials, and supplies from the site, at the completion of fieldwork. Demobilization also includes the cleanup and removal of waste generated during the investigation.

Utility Clearance

A minimum of two weeks prior to the commencement of any intrusive activities, Resolution Consultants will coordinate utility clearance with the MCRP Parris Island POC and the Palmetto Utility Protection Services (PUPS). MCRD Parris Island personnel and PUPS will identify and mark-out utilities that may be present near the proposed intrusive sampling locations. As an extra precaution, Resolution Consultants will subcontract a private utility locating vendor to identify subsurface utilities in the vicinity of proposed locations for boring and monitoring wells. The Resolution Consultants FOL will document the utility clearance process and obtain all required approvals, as deemed necessary by the MCRD Parris Island POC. Utilities that are identified in the field, but not shown or incorrectly shown on the work approval documentation, will be marked directly on the document and returned to the MCRD Parris Island POC for inclusion in the Geographic Information System database.

Equipment Calibration

Field equipment calibration procedures are described in Worksheet #22. At a minimum, field equipment will be calibrated at the beginning and end of each day, unless stated otherwise by the equipment manufacturer. Documentation of the field equipment calibration is required.

Soil Sampling

The sampling and analysis program is outlined in Worksheets #17 and #18**.** Surface and subsurface soil samples will be collected from soil borings advanced via hand tools or direct push technology tooling, in accordance with applicable U.S. EPA technical procedures, including SESDPROC-300-R2: *Soil Sampling* and SCDHEC Well Standards R.61-71, both included in Appendix A. Representative soil will be obtained, at a minimum of two foot depth intervals, for lithologic logging and field screening for organic vapors using a photo-ionization detector. Sample collection and handling will be in accordance with SESDPROC-209-R2: *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples* and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A. Samples to be analyzed for VOCs will be collected in a manner to minimize disturbance to the sample. A soil boring log will be prepared for each borings with soil descriptions and all relevant information, observations, depth to water, and field screening results. Sample depths will be included on each log. After sampling each borehole will be backfilled to within 6 inches of grade using bentonite grout in accordance with SCDHEC Well Standards R.61-71. The surface cover will be repaired using ready-mix concrete or asphalt.

Monitoring Well Installation and Development

Monitoring wells will be installed to evaluate the first (i.e., shallowest) water-bearing (water table) zone; well screens will be positioned to intersect the water table. Installation techniques, construction specifications, and well development will be in accordance with SCDHEC Well Standards R.61-71 and SESDGUID-101-R1: *Design and Installation of Monitoring Wells*, both of which are included in Appendix A.

Groundwater Level Measurements

After allowing a minimum of 24-hours equilibration time following well installation, the depth to the static water level will be measured using a water level meter in accordance with SESDPROC-105-R2: *Groundwater Level and Water Depth Measurement*, included in Appendix A. The well depth will be measured in units of feet (to the nearest 0.01 foot) with respect to the top of the polyvinyl chloride (PVC) well riser. The length of the PVC well riser will be measured from the ground surface to the reference measuring point to determine the depth-to-water below the ground surface.

Groundwater Sampling

Groundwater samples will be collected from newly installed monitoring wells in accordance with SESDPROC-301-R3: *Groundwater Sampling* and various technical procedures for water quality

monitoring and field measurements, as included in Appendix A. Sample handling will be in in accordance with SESDPROC-209-R2: *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples* and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A and as described in Worksheet #27.

Global Positioning System Locating

In accordance with SESDPROC-110-R3: *Global Positioning System*, as included in Appendix A, a hand-held GPS unit capable of sub-meter accuracy (i.e., Trimble GeoXM or Trimble GeoHX) will be used to locate sampling points. The GPS survey will utilize third order monument data, if available. Select monuments or markers (such as surveyed permanent monitoring wells) will be visited at the start and end of each day. Prior to the start of fieldwork, Resolution Consultants will load site boundaries, known cultural, or terrain features that may affect surveys, and background maps into the GPS unit. In addition, coordinates of the planned sampling locations will be loaded into the unit so that the field team can reacquire and mark each location in the field. Field personnel will have the flexibility to adjust sampling locations up to 10 feet in any direction, as necessary, to avoid physical obstructions or safety hazards (e.g., pavement, utility lines, sprinkler heads, etc.) or otherwise ensure proper placement of samples. If the Resolution Consultants FOL determines that moving a sampling location more than 10 feet is appropriate, he will contact the Resolution Consultants TOM, who will engage the NAVFAC SE RPM. Following sample collection, each location will be resurveyed using GPS to obtain the respective coordinates. GPS data collected during the survey will be stored in the GPS unit and downloaded to a computer daily or as soon as possible after acquisition. Data will also be manually entered into a field log as it is collected. Once downloaded from the GPS unit, the data will then be uploaded for processing by Resolution Consultants' Geographic Information System personnel. Certain spatial data acquired during the RI fieldwork will be uploaded as well, to ensure that significant site features are appropriately documented in the MCRD Parris Island spatial database files. To ensure sub-meter accuracy, a minimum of six satellites is desired for each position. If GPS accuracy is not sub-meter, data will not be collected until more satellites become available and the accuracy criteria are met. In locations where the overhead canopy interferes with satellite lines-of-sight and GPS accuracy cannot be reasonably established, an alternative positioning technique will be employed (e.g., compass and tape measure, fiducials, or total station) to ensure that sample locations can be reacquired at a later date, if necessary.

Land Surveying

Following installation, soil borings and monitoring wells will be surveyed by a professional surveyor licensed in the State of South Carolina. The Resolution Consultants FOL or designee will provide the surveyor with the coordinate designation nomenclature and all coordinate systems will be pre-approved prior to commencement of the survey. All measurements will be reported and recorded in U.S. Survey Feet. The surveyor will establish the horizontal location and vertical elevation for monitoring well. One horizontal measurement and two vertical measurements will be required per well. The two vertical measurements are: (1) the top of the PVC riser pipe (herein referred to as "top of casing [TOC]"), and (2) the top of ground adjacent to the pipe. All of the vertical measurements should be taken from marked positions on each temporary well or, if missing a mark, on the north side of the riser pipe. The surveyor will find and use existing survey control. It is anticipated that adequate control is located within one mile of the site. Horizontal position should be referenced to South Carolina State Plane, North American Datum 1983 and accurate to 0.10 foot. Vertical elevation should be referenced to North American Vertical Datum of 1988 and accurate to 0.01 foot. The surveyor will document all work in a clear, legible, and complete manner. The field record will contain a complete description of the nature and location of the new and existing points. The record will also include a sketch of the point locations and the benchmark witness points for both project control and local control.

Waste Handing

Solid investigative derived waste (IDW), in the form of soil, will be generated during field activities such as direct push technology sampling and well installation. Aqueous IDW will include purge water and decontamination fluids. In accordance with SESDPROC-202-R2: *Management of Investigation Derived Waste,* as included in Appendix A, IDW will be containerized in adequately labeled 55-gallon Department of Transportation-approved drums. Characterization samples will be collected and submitted for chemical analysis of Toxicity Characteristic Leaching Procedure VOCs, SVOCs, pesticides, PCBs, and metals. Pending characterization data, the drums will be temporarily staged within the site's fenced area. Based on characterization results, IDW will be transported and appropriately disposed at a South Carolina-certified offsite disposal facility. IDW disposal manifests will be appended to the forthcoming RI/RFI report.

Personal protective equipment, including gloves, wipes, discarded paper towels, and disposable equipment (i.e., tubing), will be bagged and properly discarded in an appropriate on-base solid waste dumpster.

Decontamination

Decontamination of reusable sampling equipment will be conducted prior to sampling and between samples at each location, in accordance with SESDPROC-205-R2: *Field Equipment Cleaning and Decontamination*, included as Appendix A. An area for the decontamination pad and a source of potable water for steam cleaning will be arranged by the FOL through the activity POC.

Field Documentation

Field documentation will be performed in accordance with U.S EPA technical procedures, including SESDPROC-010-R5: Logbooks and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A. A summary of field activities will be properly recorded in indelible ink in a bound logbook with consecutively numbered pages that cannot be removed. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; and the person making the correction will initial and date the change. Logbooks will be assigned to field personnel and stored in a secured area when not in use. Boring logs, sampling forms, and other field forms will also be used to document field activities.

Site Restoration

If investigation activities disturb or alter the landscape or vegetation, site surfaces will be restored to pre-existing conditions (to the extent possible). Asphalt and/or concrete patching will be performed where borings penetrate paved surfaces. Equipment and supplies used during the investigation will be removed from the site at the conclusion of field activities.

Analytical Tasks

Chemical analyses will be performed by Gulf Coast Analytical Laboratories (GCAL), a SCDHEC-accredited and DoD Environmental Laboratory Accreditation Program (ELAP)-accredited laboratory. Copies of pertinent laboratory accreditations are included in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheets #23 and #30. The subcontract laboratory will utilize screening criteria specified in Worksheet #15 and will perform the chemical analyses per laboratory-specific SOPs identified in Worksheet #23 and included in Appendix B.

Soil results will be reported by each laboratory on an adjusted dry-weight basis. Results of percent moisture will be reported in each analytical data package and associated electronic data deliverable files. This information will also be captured in the project database, which will

eventually be uploaded to the Naval Installation Restoration Information Solution database. Percent moisture information will also be captured in the RI/RFI Report.

The analytical data packages provided by GCAL will be in a Contract Laboratory Program like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method-specific quality control (QC) information (results, percent recoveries [%Rs], relative percent differences [RPDs], relative standard deviations [RSDs], percent differences or percent drifts [%Ds], etc.)

Data Management

The principal data generated for this project will be from field data and laboratory analytical data. Field sampling log sheets will be organized by date and medium, 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.

Data handling procedures to be followed by GCAL will meet the requirements of the technical specifications. Electronic data results will be automatically downloaded into the Resolution Consultants' database in accordance with the proprietary Resolution Consultants processes.

The Resolution Consultants TOM (or designee) is responsible for the overall tracking and control of data generated for the project.

Data Tracking

Data are tracked from generation to archiving in the Resolution Consultants' project-specific files. The Resolution Consultants Project Chemist (or designee) is responsible for tracking the samples collected and shipped to GCAL. Upon receipt of the data packages from GCAL, the Resolution Consultants Project Chemist will monitor data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by GCAL.

Data Storage, Archiving, and Retrieval

Data packages received from GCAL are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Resolution Consultants' Navy 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 Resolution Consultants FOL to be entered into the Navy CLEAN file system prior to archiving in secure project files. Project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Resolution Consultants.

Data Security

Access to Resolution Consultants' project files is restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Resolution Consultants Data Manager maintains the electronic data files, and access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Electronic Data

All electronic data will be compiled into a Naval Installation Restoration Information System (NIRIS) Electronic Data Deliverable and loaded into the NIRIS database.

SAP WORKSHEET #15: REFERENCE LIMITS AND EVALUATION TABLES

(UFP-QAPP Manual Section 2.8.1)

Matrix	Soil						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
1,1,1,2-Tetrachloroethane	630-20-6	0.19	RSL PG SSL RISK	0.063	**5**	**0.5**	0.178
1,1,1-Trichloroethane	71-55-6	70	RSL PG SSL MCL	23	5	0.5	0.162
1,1,2,2-Tetrachloroethane	79-34-5	0.026	RSL PG SSL RISK	0.0087	**5**	**0.5**	**0.175**
1,1,2-Trichloroethane	79-00-5	0.013	RSL PG SSL RISK	0.0043	**5**	**0.5**	**0.064**
1,1-Dichloroethane	75-34-3	0.68	RSL PG SSL RISK	0.23	**5**	0.5	0.096
1,1-Dichloroethene	75-35-4	2.5	RSL PG SSL MCL	0.83	**5**	0.5	0.227
1,1-Dichloropropene	563-58-6	NA	NA	NA	5	0.5	0.19
1,2,3-Trichlorobenzene	87-61-6	1.5	RSL PG SSL RISK	0.5	**5**	0.5	0.36
1,2,3-Trichloropropane	96-18-4	0.00028	RSL PG SSL RISK	0.000093	**5**	**0.5**	**0.291**
1,2,4-Trichlorobenzene	120-82-1	1.1	RSL PG SSL RISK	0.37	**5**	0.5	0.305
1,2,4-Trimethylbenzene	95-63-6	2.1	RSL PG SSL RISK	0.7	**5**	0.5	0.144
1,2-Dibromo-3-chloropropane	96-12-8	0.00014	RSL PG SSL RISK	0.000047	**5**	**2**	**0.285**
1,2-Dibromoethane	106-93-4	0.0018	RSL PG SSL RISK	0.0006	**5**	**2**	**0.206**
1,2-Dichlorobenzene	95-50-1	27	RSL PG SSL RISK	9	5	0.5	0.166
1,2-Dichloroethane	107-06-2	0.042	RSL PG SSL RISK	0.014	**5**	**0.5**	**0.198**
1,2-Dichloropropane	78-87-5	0.13	RSL PG SSL RISK	0.043	**5**	**0.5**	0.076
1,3,5-Trimethylbenzene	108-67-8	12	RSL PG SSL RISK	4	5	0.5	0.132
1,3-Dichlorobenzene	541-73-1	30	NOAA	10	5	0.5	0.196
1,3-Dichloropropane	142-28-9	9.9	RSL PG SSL RISK	3.3	5	0.5	0.117
1,4-Dichlorobenzene	106-46-7	0.4	RSL PG SSL RISK	0.13	**5**	**0.5**	0.2
2,2-Dichloropropane	594-20-7	NA	NA	NA	5	0.5	0.249
2-Butanone	78-93-3	100	RSL PG SSL RISK	33	5	2	0.323
2-Chlorotoluene	95-49-8	17	RSL PG SSL RISK	5.7	5	0.5	0.147
2-Hexanone	591-78-6	0.79	RSL PG SSL RISK	0.26	**5**	**2**	0.154
4-Chlorotoluene	106-43-4	18	RSL PG SSL RISK	6	5	0.5	0.154
4-Methyl-2-pentanone	108-10-1	23	RSL PG SSL RISK	7.7	5	0.5	0.154
Acetone	67-64-1	240	RSL PG SSL RISK	80	25	2	0.3

Matrix	Soil						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Benzene	71-43-2	0.2	RSL PG SSL RISK	0.067	**5**	**0.5**	0.051
Bromobenzene	108-86-1	3.6	RSL PG SSL RISK	1.2	**5**	0.5	0.152
Bromochloromethane	74-97-5	2.1	RSL PG SSL RISK	0.7	**5**	0.5	0.332
Bromodichloromethane	75-27-4	0.032	RSL PG SSL RISK	0.011	**5**	**0.5**	**0.104**
Bromoform	75-25-2	2.1	RSL PG SSL RISK	0.7	**5**	0.5	0.437
Bromomethane	74-83-9	0.18	RSL PG SSL RISK	0.06	**5**	**2**	**0.486**
Carbon disulfide	75-15-0	21	RSL PG SSL RISK	7	5	0.5	0.187
Carbon tetrachloride	56-23-5	0.15	RSL PG SSL RISK	0.05	**5**	**0.5**	**0.189**
Chlorobenzene	108-90-7	4.9	RSL PG SSL RISK	1.6	**5**	0.5	0.129
Chloroethane	75-00-3	590	RSL PG SSL RISK	200	5	0.5	0.201
Chloroform	67-66-3	0.053	RSL PG SSL RISK	0.018	**5**	**0.5**	**0.172**
Chloromethane	74-87-3	4.9	RSL PG SSL RISK	1.6	**5**	2	0.193
cis-1,2-Dichloroethene	156-59-2	0.82	RSL PG SSL RISK	0.27	**5**	0.5	0.114
cis-1,3-Dichloropropene	10061-01-5	NA	NA	NA	5	0.5	0.129
Dibromochloromethane	124-48-1	0.039	RSL PG SSL RISK	0.013	**5**	**0.5**	**0.25**
Dibromomethane	74-95-3	0.19	RSL PG SSL RISK	0.063	**5**	**0.5**	0.182
Dichlorodifluoromethane	75-71-8	30	RSL PG SSL RISK	10	5	0.5	0.224
Ethylbenzene	100-41-4	1.5	RSL PG SSL RISK	0.5	**5**	0.5	0.183
Hexachlorobutadiene	87-68-3	0.5	RSL PG SSL RISK	0.17	**5**	**0.5**	0.367
Isopropylbenzene	98-82-8	64	RSL PG SSL RISK	21	5	0.5	0.105
m- and p-Xylene	108-38-3/106-42	18	RSL PG SSL RISK	6	10	1	0.167
Methyl Tert-Butyl Ether	1634-04-4	2.8	RSL PG SSL RISK	0.93	**5**	0.5	0.061
Methylene chloride	75-09-2	1.3	RSL PG SSL MCL	0.43	**10**	1	0.512
n-Butylbenzene	104-51-8	250	RSL PG SSL RISK	83	5	0.5	0.173
Propylbenzene	103-65-1	99	RSL PG SSL RISK	33	5	0.5	0.12
o-Xylene	95-47-6	19	RSL PG SSL RISK	6.3	5	0.5	0.08
4-Isopropyltoluene	99-87-6	NA	NA	NA	5	0.5	0.119
sec-Butylbenzene	135-98-8	460	RSL PG SSL RISK	150	5	0.5	0.163
Styrene	100-42-5	100	R4 Eco Soil	33	5	0.5	0.083
tert-Butylbenzene	98-06-6	110	RSL PG SSL RISK	37	5	0.5	0.345

Matrix	Soil						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Tetrachloroethene	127-18-4	1.6	RSL PG SSL RISK	0.53	**5**	0.5	0.296
Toluene	108-88-3	50	R4 Eco Soil	17	5	0.5	0.107
trans-1,2-Dichloroethene	156-60-5	2.5	RSL PG SSL RISK	0.83	**5**	0.5	0.119
trans-1,3-Dichloropropene	10061-02-6	99	RSL PG SSL RISK	33	5	0.5	0.082
Trichloroethene	79-01-6	0.092	RSL PG SSL RISK	0.031	**5**	**0.5**	**0.127**
Trichlorofluoromethane	75-69-4	69	RSL PG SSL RISK	23	5	0.5	0.129
Vinyl chloride	75-01-4	0.0053	RSL PG SSL RISK	0.0018	**5**	**0.5**	**0.19**

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project

VOCs	=	Volatile organic compounds
CAS	=	Chemical Abstracts Service
µg/kg	=	Microgram per kilogram
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA Nov 2013)
RSL PG SSL MCL	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Maximum Contaminant Level-Based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
NA	=	Not applicable
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA, 2001).
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Soil						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
1,2,4-Trichlorobenzene	120-82-1	1.1	RSL PG SSL RISK	0.37	**330**	**33.3**	**10.5**
1,2-Diphenylhydrazine	122-66-7	0.22	RSL PG SSL RISK	0.073	**330**	**16.7**	**6.8**
2,4,5-Trichlorophenol	95-95-4	330	RSL PG SSL RISK	110	330	66.7	10.4
2,4,6-Trichlorophenol	88-06-2	3.4	RSL PG SSL RISK	1.1	**330**	**167**	**11.6**
2,4-Dichlorophenol	120-83-2	4.1	RSL PG SSL RISK	1.4	**330**	**66.7**	**9.5**
2,4-Dimethylphenol	105-67-9	10	NOAA	3.3	**330**	**330**	7
2,4-Dinitrophenol	51-28-5	3.4	RSL PG SSL RISK	1.1	**1650**	**330**	**23**
2,4-Dinitrotoluene	121-14-2	0.28	RSL PG SSL RISK	0.093	**330**	**66.7**	**7**
2,6-Dichlorophenol	87-65-0	10	NOAA	3.3	**330**	**33.3**	4.2
2,6-Dinitrotoluene	606-20-2	0.058	RSL PG SSL RISK	0.019	**330**	**33.3**	**12.3**
2-Chloronaphthalene	91-58-7	290	RSL PG SSL RISK	97	**330**	33.3	10.5
2-Chlorophenol	95-57-8	5.7	RSL PG SSL RISK	1.9	**330**	**33.3**	**7.2**
2-Methylphenol	95-48-7	50	NOAA	17	**330**	33.3	10.2
2-Nitroaniline	88-74-4	6.2	RSL PG SSL RISK	2.1	**1650**	**66.7**	4
2-Nitrophenol	88-75-5	1600	NOAA	530	330	33.3	11.8
3,3-Dichlorobenzidine	91-94-1	0.71	RSL PG SSL RISK	0.24	**660**	**330**	**32**
3- and 4-Methylphenol	108-39-4/106-44	50	NOAA	17	**330**	**167**	12.4
3-Nitroaniline	99-09-2	NA	NA	NA	1650	66.7	4
4,6-Dinitro-2-methylphenol	534-52-1	0.2	RSL PG SSL RISK	0.067	**1650**	**330**	**18**
4-Bromophenyl-phenylether	101-55-3	NA	NA	NA	330	33.3	10.5
4-Chloro-3-methylphenol	59-50-7	130	RSL PG SSL RISK	43	**330**	66.7	7.4
4-Chloroaniline	106-47-8	0.13	RSL PG SSL RISK	0.043	**330**	**33.3**	**6.3**
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	NA	330	33.3	7.3
4-Nitroaniline	100-01-6	1.4	RSL PG SSL RISK	0.47	**1650**	**330**	**4**
4-Nitrophenol	100-02-7	700	R4 Eco Soil	230	**1650**	330	21
Benzidine	92-87-5	0.00024	RSL PG SSL RISK	0.00008	**1650**	**660**	**564**
Benzoic acid	65-85-0	1400	RSL PG SSL RISK	470	**1650**	167	21
Benzyl alcohol	100-51-6	37	RSL PG SSL RISK	12	**330**	**167**	10.8
Bis(2-chloroethoxy)methane	111-91-1	1.1	RSL PG SSL RISK	0.37	**330**	**33.3**	**5.8**
Bis(2-chloroethyl)ether	111-44-4	0.0031	RSL PG SSL RISK	0.001	**330**	**33.3**	**12.8**

Matrix	Soil						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Bis(2-chloroisopropyl)ether	39638-32-9	NA	NA	NA	330	33.3	7.2
Bis(2-ethylhexyl)phthalate	117-81-7	100	NOAA	33	**330**	33.3	4.7
Butylbenzylphthalate	85-68-7	100	NOAA	33	**330**	16.7	6.6
Carbazole	86-74-8	NA	NA	NA	330	33.3	6
Dibenzofuran	132-64-9	11	RSL PG SSL RISK	3.7	**330**	**33.3**	6.3
Diethylphthalate	84-66-2	470	RSL PG SSL RISK	160	330	33.3	12.2
Dimethyl phthalate	131-11-3	200000	R4 Eco Soil	67000	330	16.7	5.1
Di-n-butylphthalate	84-74-2	170	RSL PG SSL RISK	57	**330**	16.7	4.4
Di-n-octylphthalate	117-84-0	100	NOAA	33	**330**	16.7	4.4
Hexachlorobenzene	118-74-1	0.53	RSL PG SSL RISK	0.18	**330**	**66.7**	**12.8**
Hexachloroethane	67-72-1	0.31	RSL PG SSL RISK	0.1	**330**	**33.3**	**15.7**
Isophorone	78-59-1	22	RSL PG SSL RISK	7.3	**330**	**33.3**	5.9
Nitrobenzene	98-95-3	0.079	RSL PG SSL RISK	0.026	**330**	**33.3**	**17.2**
N-Nitrosodinpropylamine	621-64-7	0.007	RSL PG SSL RISK	0.0023	**330**	**33.3**	**14.4**
N-Nitrosodiphenylamine	86-30-6	57	RSL PG SSL RISK	19	**330**	33.3	7
N-Nitrosodimethylamine	62-75-9	0.0001	RSL PG SSL RISK	0.000033	**330**	**66.7**	**11.6**
Pentachlorophenol	87-86-5	0.36	RSL PG SSL RISK	0.12	**1650**	**330**	**7**
Phenol	108-95-2	50	R4 Eco Soil	17	**330**	33.3	9.1
2-Methylnaphthalene	91-57-6	14	RSL PG SSL RISK	4.7	3.3	1.7	0.698
Acenaphthene	83-32-9	410	RSL PG SSL RISK	140	3.3	1.7	1.09
Acenaphthylene	208-96-8	NA	NA	NA	3.3	1.7	1.31
Anthracene	120-12-7	100	R4 Eco Soil	33	3.3	1.7	0.269
Benzo[a]anthracene	56-55-3	10	RSL PG SSL RISK	3.3	3.3	1.7	1.54
Benzo[a]pyrene	50-32-8	3.5	RSL PG SSL RISK	1.2	3.3	1.7	0.31
Benzo[b]fluoranthene	205-99-2	35	RSL PG SSL RISK	12	3.3	1.7	0.988
Benzo[g,h,i]perylene	191-24-2	NA	NA	NA	3.3	1.7	0.595
Benzo[k]fluoranthene	207-08-9	350	RSL PG SSL RISK	120	3.3	1.7	0.388
Chrysene	218-01-9	1100	RSL PG SSL RISK	370	3.3	1.7	1.11
Dibenz[a,h]anthracene	53-70-3	11	RSL PG SSL RISK	3.7	3.3	1.7	0.99
Fluoranthene	206-44-0	100	R4 Eco Soil	33	3.3	1.7	0.366

Matrix	Soil						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Fluorene	86-73-7	400	RSL PG SSL RISK	130	3.3	1.7	0.408
Indeno[1,2,3-cd]pyrene	193-39-5	150	RSL PG SSL RISK	50	3.3	1.7	0.421
Naphthalene	91-20-3	0.47	RSL PG SSL RISK	0.16	**3.3**	**1.7**	0.372
Phenanthrene	85-01-8	100	R4 Eco Soil	33	3.3	1.7	0.474
Pyrene	129-00-0	100	R4 Eco Soil	33	3.3	1.7	0.552

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

SVOCs	=	Semi-volatile organic compounds
CAS	=	Chemical Abstracts Service
µg/kg	=	Microgram per kilogram
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA 2001).
NA	=	Not applicable
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Soil						
Analytical Group	Pesticides						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
4,4-DDD	72-54-8	2.5	R4 Eco Soil	0.83	**3.33**	2	0.735
4,4-DDE	72-55-9	2.5	R4 Eco Soil	0.83	**3.33**	2	0.351
4,4-DDT	50-29-3	2.5	R4 Eco Soil	0.83	**3.33**	2	0.993
Aldrin	309-00-2	0.65	RSL PG SSL RISK	0.22	**1.67**	**2**	0.347
alpha-BHC	319-84-6	0.036	RSL PG SSL RISK	0.012	**1.67**	**2**	**0.5**
alpha-Chlordane	5103-71-9	0.03	NOAA	0.01	**1.67**	**2**	**0.47**
beta-BHC	319-85-7	0.13	RSL PG SSL RISK	0.043	**1.67**	**2**	**0.439**
delta-BHC	319-86-8	NA	NA	NA	1.67	2	0.412
Dieldrin	60-57-1	0.061	RSL PG SSL RISK	0.02	**3.33**	**2**	**0.365**
Endosulfan I	959-98-8	0.01	NOAA	0.0033	**1.67**	**2**	**0.361**
Endosulfan II	33213-65-9	0.01	NOAA	0.0033	**3.33**	**2**	**0.445**
Endosulfan sulfate	1031-07-8	35.8	NOAA	12	3.33	2	0.544
Endrin	72-20-8	1	R4 Eco Soil	0.33	**3.33**	**2**	0.706
Endrin aldehyde	7421-93-4	10.5	NOAA	3.5	3.33	2	0.627
Endrin ketone	53494-70-5	NA	NA	NA	3.33	2	0.831
Lindane	58-89-9	0.05	R4 Eco Soil	0.017	**1.67**	**2**	**0.514**
gamma-Chlordane	5566-34-7	0.03	NOAA	0.01	**1.67**	**2**	**0.638**
Heptachlor	76-44-8	0.14	RSL PG SSL RISK	0.047	**1.67**	**2**	**0.424**
Heptachlor epoxide	1024-57-3	0.0002	NOAA	0.000067	**1.67**	**2**	**0.399**
Methoxychlor	72-43-5	19.9	NOAA	6.6	1.67	2	1.38

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

CAS	=	Chemical Abstracts Service
µg/kg	=	Microgram per kilogram
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA, 2001).
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)

NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Soil						
Analytical Group	PCBs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Aroclor-1016	12674-11-2	10	RSL PG SSL RISK	3.3	**33.3**	**17**	**13.3**
Aroclor-1221	11104-28-2	0.069	RSL PG SSL RISK	0.023	**33.3**	**17**	**13.3**
Aroclor-1232	11141-16-5	0.069	RSL PG SSL RISK	0.023	**33.3**	**17**	**13.3**
Aroclor-1242	53469-21-9	5.3	RSL PG SSL RISK	1.8	**33.3**	**17**	**13.3**
Aroclor-1248	12672-29-6	5.2	RSL PG SSL RISK	1.7	**33.3**	**17**	**13.3**
Aroclor-1254	11097-69-1	8.2	RSL PG SSL RISK	2.7	**33.3**	**17**	**13.3**
Aroclor-1260	11096-82-5	20	R4 Eco Soil	6.7	**33.3**	17	13.3
Aroclor-1262	37324-23-5	20	R4 Eco Soil	6.7	**33.3**	17	13.3
Aroclor-1268	11100-14-4	20	R4 Eco Soil	6.7	**33.3**	17	13.3

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

PCBs	=	Polychlorinated biphenyl compounds
CAS	=	Chemical Abstracts Service
µg/kg	=	Microgram per kilogram
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA, 2001).
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Soil						
Analytical Group	Metals						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/kg)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/kg)**	**LOQ (µg/kg)**	**LOD (µg/kg)**	**DL (µg/kg)**
Aluminum	7429-90-5	50000	R4 Eco Soil	17000	800	400	200
Antimony	7440-36-0	27	RSL PG SSL RISK	9	**80**	**40**	20
Arsenic	7440-38-2	1.3	RSL PG SSL RISK	0.43	**40**	**20**	**10**
Barium	7440-39-3	12000	RSL PG SSL RISK	4000	40	20	10
Beryllium	7440-41-7	1100	R4 Eco Soil	370	40	20	10
Cadmium	7440-43-9	52	RSL PG SSL RISK	17	40	20	10
Calcium	7440-70-2	NA	NA	NA	20000	10000	5000
Chromium, Total	7440-47-3	400	R4 Eco Soil	130	40	20	10
Hexavalent Chromium	18540-29-9	0.59	RSL PG SSL RISK	0.2	**500**	**500**	**310**
Cobalt	7440-48-4	21	RSL PG SSL RISK	7	**40**	20	10
Copper	7440-50-8	2200	RSL PG SSL RISK	730	40	20	10
Iron	7439-89-6	27000	RSL PG SSL RISK	9000	4000	2000	1000
Lead	7439-92-1	14000	RSL PG SSL MCL	4700	40	20	10
Magnesium	7439-95-4	NA	NA	NA	4000	2000	1000
Manganese	7439-96-5	2100	RSL PG SSL RISK	700	200	100	50
Mercury	7439-97-6	3.3	RSL PG SSL RISK	1.1	**10**	**10**	**4**
Molybdenum	7439-98-7	160	RSL PG SSL RISK	53	40	20	10
Nickel	7440-02-0	2000	RSL PG SSL RISK	670	80	40	20
Potassium	9/7/7440	NA	NA	NA	4000	2000	1000
Selenium	7782-49-2	40	RSL PG SSL RISK	13	**40**	20	10
Silver	7440-22-4	60	RSL PG SSL RISK	20	40	20	10
Sodium	7440-23-5	NA	NA	NA	4000	2000	1000
Thallium	7440-28-0	1.1	RSL PG SSL RISK	0.37	**40**	**20**	**10**
Vanadium	7440-62-2	2000	R4 Eco Soil	670	40	20	10
Zinc	7440-66-6	29000	RSL PG SSL RISK	9700	40	20	10

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.
CAS = Chemical Abstracts Service
µg/kg = Microgram per kilogram
LOQ = Limit of quantification

LOD	=	Limit of detection
DL	=	Detection Limit
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA, 2001).
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
NA	=	Not applicable
RSL PG SSL MCL	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Maximum Contaminant Level-Based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Groundwater						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
1,1,1,2-Tetrachloroethane	630-20-6	0.5	RSL GW TAP	0.17	**1**	0.2	0.12
1,1,1-Trichloroethane	71-55-6	200	RSL GW MCL	67	1	0.2	0.123
1,1,2,2-Tetrachloroethane	79-34-5	0.066	RSL GW TAP	0.022	**1**	**0.2**	**0.109**
1,1,2-Trichloroethane	79-00-5	0.041	RSL GW TAP	0.014	**1**	**0.2**	**0.159**
1,1-Dichloroethane	75-34-3	2.4	RSL GW TAP	0.8	1	0.2	0.171
1,1-Dichloroethene	75-35-4	7	RSL GW MCL	2.3	1	0.5	0.2
1,1-Dichloropropene	563-58-6	NA	NA	NA	1	0.2	0.052
1,2,3-Trichlorobenzene	87-61-6	0.52	RSL GW TAP	0.17	**1**	0.2	0.079
1,2,3-Trichloropropane	96-18-4	0.00065	RSL GW TAP	0.00022	**1**	**0.2**	**0.065**
1,2,4-Trichlorobenzene	120-82-1	0.39	RSL GW TAP	0.13	**1**	0.2	0.105
1,2,4-Trimethylbenzene	95-63-6	1.5	RSL GW TAP	0.5	1	0.2	0.084
1,2-Dibromo-3-chloropropane	96-12-8	0.00032	RSL GW TAP	0.00011	**1**	**0.2**	**0.194**
1,2-Dibromoethane	106-93-4	0.0065	RSL GW TAP	0.0022	**1**	**0.2**	**0.102**
1,2-Dichlorobenzene	95-50-1	19.7	R4 Eco SW	6.6	1	0.2	0.135
1,2-Dichloroethane	107-06-2	0.15	RSL GW TAP	0.05	**1**	**0.2**	0.116
1,2-Dichloropropane	78-87-5	0.38	RSL GW TAP	0.13	**1**	0.2	0.15
1,3,5-Trimethylbenzene	108-67-8	8.7	RSL GW TAP	2.9	1	0.2	0.066
1,3-Dichlorobenzene	541-73-1	28.5	R4 Eco SW	9.5	1	0.2	0.138
1,3-Dichloropropane	142-28-9	29	RSL GW TAP	9.7	1	0.2	0.059
1,4-Dichlorobenzene	106-46-7	0.42	RSL GW TAP	0.14	**1**	0.2	0.083
2,2-Dichloropropane	594-20-7	NA	NA	NA	1	0.2	0.17
2-Butanone	78-93-3	490	RSL GW TAP	160	5	0.5	0.142
2-Chlorotoluene	95-49-8	18	RSL GW TAP	6	1	0.2	0.08
2-Hexanone	591-78-6	3.4	RSL GW TAP	1.1	**5**	1	0.122
4-Chlorotoluene	106-43-4	19	RSL GW TAP	6.3	1	0.2	0.124
4-Methyl-2-pentanone	108-10-1	100	RSL GW TAP	33	5	0.5	0.12
Acetone	67-64-1	1200	RSL GW TAP	400	5	1	0.193
Benzene	71-43-2	0.39	RSL GW TAP	0.13	**1**	0.2	0.111
Bromobenzene	108-86-1	5.4	RSL GW TAP	1.8	1	0.2	0.145
Bromochloromethane	74-97-5	8.3	RSL GW TAP	2.8	1	0.5	0.127

Matrix	Groundwater						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
Bromodichloromethane	75-27-4	0.12	RSL GW TAP	0.04	**1**	**0.2**	0.083
Bromoform	75-25-2	7.9	RSL GW TAP	2.6	1	0.5	0.215
Bromomethane	74-83-9	0.7	RSL GW TAP	0.23	**1**	0.5	0.427
Carbon disulfide	75-15-0	0.72	RSL GW TAP	0.24	**1**	0.5	0.19
Carbon tetrachloride	56-23-5	0.39	RSL GW TAP	0.13	**1**	**0.5**	0.248
Chlorobenzene	108-90-7	7.2	RSL GW TAP	2.4	1	0.2	0.083
Chloroethane	75-00-3	2100	RSL GW TAP	700	1	0.5	0.235
Chloroform	67-66-3	0.19	RSL GW TAP	0.063	**1**	**0.2**	0.155
Chloromethane	74-87-3	19	RSL GW TAP	6.3	1	0.2	0.144
cis-1,2-Dichloroethene	156-59-2	2.8	RSL GW TAP	0.93	1	0.2	1.03
cis-1,3-Dichloropropene	10061-01-5	290	RSL GW TAP-surr	97	1	0.2	0.124
Dibromochloromethane	124-48-1	0.15	RSL GW TAP	0.05	**1**	**0.2**	0.054
Dibromomethane	74-95-3	0.79	RSL GW TAP	0.26	**1**	0.5	0.211
Dichlorodifluoromethane	75-71-8	19	RSL GW TAP	6.3	1	0.2	0.145
Ethylbenzene	100-41-4	1.3	RSL GW TAP	0.43	1	0.2	0.109
Hexachlorobutadiene	87-68-3	0.26	RSL GW TAP	0.087	**5**	**1**	**0.265**
Isopropylbenzene	98-82-8	39	RSL GW TAP	13	1	0.2	0.13
m- and p-Xylene	108-38-3/106-42	19	RSL GW TAP	6.3	2	0.4	0.123
Methyl Tert-Butyl Ether	1634-04-4	12	RSL GW TAP	4	1	0.2	0.078
Methylene chloride	75-09-2	5	RSL GW MCL	1.7	**5**	0.5	0.149
n-Butylbenzene	104-51-8	78	RSL GW TAP	26	1	0.2	0.123
Propylbenzene	103-65-1	53	RSL GW TAP	18	1	1	0.727
o-Xylene	95-47-6	19	RSL GW TAP	6.3	1	0.2	0.055
4-Isopropyltoluene	99-87-6	NA	NA	NA	1	0.2	0.07
sec-Butylbenzene	135-98-8	160	RSL GW TAP	53	1	0.2	0.107
Styrene	100-42-5	100	RSL GW MCL	33	1	0.2	0.089
tert-Butylbenzene	98-06-6	51	RSL GW TAP	17	1	0.2	0.087
Tetrachloroethene	127-18-4	3.5	RSL GW TAP	1.2	1	0.5	0.193
Toluene	108-88-3	37	R4 Eco SW	12	1	0.2	0.122
trans-1,2-Dichloroethene	156-60-5	8.6	RSL GW TAP	2.9	1	0.2	0.077

Matrix	Groundwater						
Analytical Group	VOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
trans-1,3-Dichloropropene	10061-02-6	290	RSL GW TAP-surr	97	1	0.2	0.128
Trichloroethene	79-01-6	0.26	RSL GW TAP	0.087	**1**	0.2	0.161
Trichlorofluoromethane	75-69-4	110	RSL GW TAP	37	1	0.2	0.157
Vinyl chloride	75-01-4	0.015	RSL GW TAP	0.005	**1**	**0.2**	**0.127**

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

VOCs	=	Volatile organic compounds
CAS	=	Chemical Abstracts Service
µg/L	=	Microgram per liter
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL GW TAP	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Tap Water (U.S. EPA Nov 2013)
RSL GW MCL	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Maximum Contaminant Levels (U.S. EPA Nov 2013)
NA	=	Not applicable
R4 Eco SW	=	U.S. EPA Region 4 Ecological Surface Water Screening Level (U.S. EPA 2001).
Surr	=	Structurally similar surrogate compound used
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Groundwater						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
1,2,4-Trichlorobenzene	120-82-1	0.39	RSL GW TAP	0.13	**10**	**0.5**	**0.468**
1,2-Diphenylhydrazine	122-66-7	0.067	RSL GW TAP	0.022	**10**	**0.5**	**0.378**
2,4,5-Trichlorophenol	95-95-4	11	NOAA	3.7	10	0.5	0.269
2,4,6-Trichlorophenol	88-06-2	0.9	RSL GW TAP	0.3	**10**	0.5	0.324
2,4-Dichlorophenol	120-83-2	3.5	RSL GW TAP	1.2	**10**	0.5	0.383
2,4-Dimethylphenol	105-67-9	27	RSL GW TAP	9	10	1.2	0.37
2,4-Dinitrophenol	51-28-5	3	RSL GW TAP	1	**50**	**5**	1.4
2,4-Dinitrotoluene	121-14-2	0.2	RSL GW TAP	0.067	**10**	**0.5**	**0.283**
2,6-Dichlorophenol	87-65-0	NA	NA	NA	10	0.5	0.444
2,6-Dinitrotoluene	606-20-2	0.042	RSL GW TAP	0.014	**10**	**0.5**	**0.246**
2-Chloronaphthalene	91-58-7	55	RSL GW TAP	18	10	0.5	0.391
2-Chlorophenol	95-57-8	7.1	RSL GW TAP	2.4	**10**	0.5	0.309
2-Methylphenol	95-48-7	72	RSL GW TAP	24	10	0.5	0.253
2-Nitroaniline	88-74-4	15	RSL GW TAP	5	10	0.5	0.154
2-Nitrophenol	88-75-5	4500	RSL GW TAP-surr	1500	10	0.5	0.457
3,3-Dichlorobenzidine	91-94-1	0.11	RSL GW TAP	0.037	**10**	**0.5**	**0.122**
3- and 4-Methylphenol	108-39-4/106-44	72	RSL GW TAP	24	10	0.5	0.177
3-Nitroaniline	99-09-2	3.3	R4 Eco SW	1.1	**50**	0.5	0.304
4,6-Dinitro-2-methylphenol	534-52-1	0.12	RSL GW TAP	0.04	**50**	**5**	**1.07**
4-Bromophenyl-phenylether	101-55-3	NA	NA	NA	10	0.5	0.451
4-Chloro-3-methylphenol	59-50-7	110	RSL GW TAP	37	10	0.5	0.332
4-Chloroaniline	106-47-8	0.32	RSL GW TAP	0.11	**10**	**0.5**	0.291
4-Chlorophenyl-phenylether	7005-72-3	NA	NA	NA	10	0.5	0.398
4-Nitroaniline	100-01-6	3.3	RSL GW TAP	1.1	**50**	**5**	0.328
4-Nitrophenol	100-02-7	71.7	R4 Eco SW	24	50	5	1.17
Benzidine	92-87-5	0.000092	RSL GW TAP	0.000031	**50**	**5**	**2.12**
Benzoic acid	65-85-0	5800	RSL GW TAP	1900	50	1.2	1.13
Benzyl alcohol	100-51-6	150	RSL GW TAP	50	10	0.5	0.429
Bis(2-chloroethoxy)methane	111-91-1	4.6	RSL GW TAP	1.5	**10**	0.5	0.405
Bis(2-chloroethyl)ether	111-44-4	0.012	RSL GW TAP	0.004	**10**	**0.5**	**0.422**

Matrix	Groundwater						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
Bis(2-chloroisopropyl)ether	39638-32-9	NA	NA	NA	10	0.5	0.406
Bis(2-ethylhexyl)phthalate	117-81-7	4.8	RSL GW TAP	1.6	**10**	0.5	0.186
Butylbenzylphthalate	85-68-7	14	RSL GW TAP	4.7	10	5	0.213
Carbazole	86-74-8	NA	NA		10	0.5	0.264
Dibenzofuran	132-64-9	0.58	RSL GW TAP	0.19	**10**	0.5	0.371
Diethylphthalate	84-66-2	75.9	R4 Eco SW	25	10	0.5	0.262
Dimethyl phthalate	131-11-3	NA	NA	NA	10	0.5	0.336
Di-n-butylphthalate	84-74-2	3.4	R4 Eco SW	1.1	**10**	0.5	0.262
Di-n-octylphthalate	117-84-0	16	RSL GW TAP	5.3	10	0.5	0.123
Hexachlorobenzene	118-74-1	0.042	RSL GW TAP	0.014	**10**	**1.2**	**0.499**
Hexachloroethane	67-72-1	0.51	RSL GW TAP	0.17	**10**	**1.2**	0.476
Isophorone	78-59-1	67	RSL GW TAP	22	10	0.5	0.368
Nitrobenzene	98-95-3	0.12	RSL GW TAP	0.04	**10**	**0.5**	**0.381**
N-Nitrosodinpropylamine	621-64-7	0.0093	RSL GW TAP	0.0031	**10**	**0.5**	**0.417**
N-Nitrosodiphenylamine	86-30-6	10	RSL GW TAP	3.3	**10**	0.5	0.335
N-Nitrosodimethylamine	62-75-9	0.00042	RSL GW TAP	0.00014	**10**	**0.5**	**0.248**
Pentachlorophenol	87-86-5	0.035	RSL GW TAP	0.012	**50**	**0.5**	**0.288**
Phenol	108-95-2	58	R4 Eco SW	19	10	0.5	0.19
2-Methylnaphthalene	91-57-6	2.7	RSL GW TAP	0.9	0.1	0.1	0.0296
Acenaphthene	83-32-9	9.7	R4 Eco SW	3.2	0.1	0.1	0.0082
Acenaphthylene	208-96-8	400	RSL GW TAP-surr	130	0.1	0.1	0.0253
Anthracene	120-12-7	130	RSL GW TAP	43	0.1	0.1	0.041
Benzo[a]anthracene	56-55-3	0.029	RSL GW TAP	0.0097	**0.1**	**0.1**	0.0197
Benzo[a]pyrene	50-32-8	0.0029	RSL GW TAP	0.00097	**0.1**	**0.1**	**0.0114**
Benzo[b]fluoranthene	205-99-2	0.029	RSL GW TAP	0.0097	**0.1**	**0.1**	0.0087
Benzo[g,h,i]perylene	191-24-2	87	RSL GW TAP-surr	29	0.1	0.1	0.011
Benzo[k]fluoranthene	207-08-9	0.29	RSL GW TAP	0.097	0.1	0.1	0.0166
Chrysene	218-01-9	2.9	RSL GW TAP	0.97	0.1	0.1	0.0176
Dibenz[a,h]anthracene	53-70-3	0.0029	RSL GW TAP	0.00097	**0.1**	**0.1**	**0.016**
Fluoranthene	206-44-0	1.6	R4 Eco SW	0.53	0.1	0.1	0.012

Matrix	Groundwater						
Analytical Group	SVOCs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
Fluorene	86-73-7	22	RSL GW TAP	7.3	0.1	0.1	0.0397
Indeno[1,2,3-cd]pyrene	193-39-5	0.029	RSL GW TAP	0.0097	**0.1**	**0.1**	0.0052
Naphthalene	91-20-3	0.14	RSL GW TAP	0.047	0.1	0.1	0.0097
Phenanthrene	85-01-8	4.6	NOAA	1.5	0.1	0.1	0.0072
Pyrene	129-00-0	8.7	RSL GW TAP	2.9	0.1	0.1	0.0148

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

SVOCs	=	Semi-volatile organic compounds
CAS	=	Chemical Abstracts Service
µg/L	=	Microgram per liter
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL GW TAP	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Tap Water (U.S. EPA Nov 2013)
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
NA	=	Not applicable
Surr	=	Structurally similar surrogate compound used
R4 Eco SW	=	U.S. EPA Region 4 Ecological Surface Water Screening Level (U.S. EPA 2001).
Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Groundwater						
Analytical Group	Pesticides						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
4,4-DDD	72-54-8	0.025	R4 Eco SW	0.0083	**0.1**	0.01	0.0013
4,4-DDE	72-55-9	0.14	R4 Eco SW	0.047	0.1	0.01	0.001
4,4-DDT	50-29-3	0.001	R4 Eco SW	0.00033	**0.1**	**0.01**	**0.0023**
Aldrin	309-00-2	0.004	RSL GW TAP	0.0013	**0.05**	**0.01**	0.0019
alpha-BHC	319-84-6	0.0062	RSL GW TAP	0.0021	**0.03**	**0.01**	0.0014
alpha-Chlordane	5103-71-9	0.004	R4 Eco SW	0.0013	**0.05**	**1**	0.0021
beta-BHC	319-85-7	0.022	RSL GW TAP	0.0073	**0.05**	0.01	0.0011
delta-BHC	319-86-8	NA	NA	NA	0.05	0.01	0.0012
Dieldrin	60-57-1	0.0015	RSL GW TAP	0.0005	**0.1**	**0.01**	**0.0015**
Endosulfan I	959-98-8	0.00435	NOAA	0.0015	**0.05**	**0.01**	0.002
Endosulfan II	33213-65-9	0.00435	NOAA	0.0015	**0.1**	**0.01**	0.0024
Endosulfan sulfate	1031-07-8	NA	NA	NA	0.1	0.01	0.002
Endrin	72-20-8	0.0023	R4 Eco SW	0.00077	**0.1**	**0.01**	0.0012
Endrin aldehyde	7421-93-4	NA	NA	NA	0.1	0.01	0.0035
Endrin ketone	53494-70-5	NA	NA	NA	0.1	0.01	0.0015
Lindane	58-89-9	0.016	R4 Eco SW	0.0053	**0.05**	0.01	0.001
gamma-Chlordane	5566-34-7	NA	NA	NA	0.05	0.01	0.001
Heptachlor	76-44-8	0.0018	RSL GW TAP	0.0006	**0.05**	**0.01**	**0.0037**
Heptachlor epoxide	1024-57-3	0.0033	RSL GW TAP	0.0011	**0.05**	**0.01**	0.001
Methoxychlor	72-43-5	0.03	R4 Eco SW	0.01	**0.5**	0.02	0.0022

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

CAS	=	Chemical Abstracts Service
µg/L	=	Microgram per liter
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
R4 Eco SW	=	U.S. EPA Region 4 Ecological Surface Water Screening Level (U.S. EPA 2001).
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL GW TAP	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Tap Water (U.S. EPA Nov 2013)
NA	=	Not appliable
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables

Bolded	=	PAL are less than the laboratory LOQ; however, the LOD is sufficiently low to meet the PAL and for the intended data use.
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Groundwater						
Analytical Group	PCBs						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
Aroclor-1016	12674-11-2	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1221	11104-28-2	0.004	RSL GW TAP	0.0013	**0.5**	**0.5**	**0.4**
Aroclor-1232	11141-16-5	0.004	RSL GW TAP	0.0013	**0.5**	**0.5**	**0.4**
Aroclor-1242	53469-21-9	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1248	12672-29-6	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1254	11097-69-1	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1260	11096-82-5	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1262	37324-23-5	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**
Aroclor-1268	11100-14-4	0.03	R4 Eco SW	0.01	**0.5**	**0.5**	**0.4**

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.

PCBs	=	Polychlorinated biphenyl compounds
CAS	=	Chemical Abstracts Service
µg/L	=	Microgram per liter
LOQ	=	Limit of quantification
LOD	=	Limit of detection
DL	=	Detection Limit
R4 Eco SW	=	U.S. EPA Region 4 Ecological Surface Water Screening Level (U.S. EPA 2001).
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL GW TAP	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Tap Water (U.S. EPA Nov 2013)
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

Matrix	Groundwater						
Analytical Group	Metals						
					Laboratory Specific Limits [1]		
Analyte	**CAS No.**	**Project Action Level (µg/L)**	**Project Action Level Reference**	**Project Quantitation Limit Goal (µg/L)**	**LOQ (µg/L)**	**LOD (µg/L)**	**DL (µg/L)**
Aluminum	7429-90-5	1600	RSL GW TAP	530	20	10	5
Antimony	7440-36-0	0.6	RSL GW TAP	0.2	**2**	**1**	0.5
Arsenic	7440-38-2	0.045	RSL GW TAP	0.015	**1**	**0.5**	**0.25**
Barium	7440-39-3	200	NOAA	67	1	0.5	0.25
Beryllium	7440-41-7	1.6	RSL GW TAP	0.53	1	0.5	0.25
Cadmium	7440-43-9	NA	NA	NA	1	0.5	0.25
Calcium	7440-70-2	NA	NA	NA	500	250	125
Chromium, Total	7440-47-3	100	RSL GW MCL	33	1	0.5	0.25
Cobalt	7440-48-4	0.47	RSL GW TAP	0.16	**1**	**0.5**	0.25
Copper	7440-50-8	2.9	R4 Eco SW	0.97	1	0.5	0.25
Iron	7439-89-6	1100	RSL GW TAP	370	100	50	25
Lead	7439-92-1	8.5	R4 Eco SW	2.8	1	0.5	0.25
Magnesium	7439-95-4	NA	NA	NA	100	50	25
Manganese	7439-96-5	100	NOAA	33	5	3	1.25
Mercury	7439-97-6	0.025	R4 Eco SW	0.0083	**0.2**	**0.2**	**0.07**
Molybdenum	7439-98-7	7.8	RSL GW TAP	2.6	1	0.5	0.25
Nickel	7440-02-0	3	RSL GW TAP	1	2	1	0.5
Potassium	9/7/7440	NA	NA	NA	100	50	25
Selenium	7782-49-2	7.8	RSL GW TAP	2.6	1	0.5	0.25
Silver	7440-22-4	0.23	R4 Eco SW	0.077	**1**	**0.5**	**0.25**
Sodium	7440-23-5	NA	NA	NA	100	50	25
Thallium	7440-28-0	0.016	RSL GW TAP	0.0053	**1**	**0.5**	**0.25**
Vanadium	7440-62-2	6.3	RSL GW TAP	2.1	1	0.5	0.25
Zinc	7440-66-6	86	R4 Eco SW	29	20	10	5

Notes:
[1] LOQ, LOD, and DLs are provided by Gulf Coast Analytical Laboratories and may vary during the course of project.
CAS = Chemical Abstracts Service
µg/L = Microgram per liter
LOQ = Limit of quantification
LOD = Limit of detection

DL	=	Detection Limit
RSL	=	Regional Screening Levels
U.S. EPA	=	U.S. Environmental Protection Agency
RSL GW TAP	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Tap Water (U.S. EPA Nov 2013)
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
NA	=	Not applicable
RSL GW MCL	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites – Maximum Contaminant Levels (U.S. EPA Nov 2013)
R4 Eco SW	=	U.S. EPA Region 4 Ecological Surface Water Screening Level (U.S. EPA 2001).
Shaded	=	LOQs and LODs do not meet the PAL. Uncertainties introduced by LODs or LOQs that are greater than PALs will be described in the Remedial Investigation Report. The DL is provided for completeness of evaluation.

SAP WORKSHEET #16: PROJECT/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/Submit Draft SAP	Resolution Consultants	11/26/12	5/3/13	Draft SAP	5/3/13
Regulatory Review	U.S. EPA, SCDHEC	5/3/13	12/18/13	NA	NA
Prepare/Submit Draft Final SAP	Resolution Consultants	12/18/13	5/30/14	Draft Final SAP	5/30/14
Regulatory Review	U.S. EPA, SCDHEC	5/30/14	6/29/14	NA	NA
Prepare/Submit Final SAP	Resolution Consultants	6/29/14	7/14/14	Final SAP	7/14/14
Concurrence by Regulators	U.S. EPA, SCDHEC	7/14/14	7/28/14	NA	NA
Utility Clearance	Resolution Consultants, MCRD Parris Island	7/28/14	8/1/14	NA	NA
Soil Sampling	Resolution Consultants	8/4/14	8/7/14	NA	NA
Well Installation and Development	Resolution Consultants	8/8/14	8/13/14	NA	NA
Groundwater Measurement/Sampling/Survey	Resolution Consultants	8/14/14	8/15/14	NA	NA
Laboratory Results	GCAL	8/15/14	9/14/14	Laboratory data packages and electronic data deliverables	9/14/14
Prepare/Submit Internal Draft RI Report	Resolution Consultants	9/14/14	10/26/14	Internal Draft RI Report	10/26/14
Navy Review of RI Report Complete	Navy	10/26/14	11/9/14	NA	NA
Prepare/Submit Draft RI Report	Resolution Consultants	11/9/14	12/1/14	Draft RI Report	12/1/14
Regulatory Review	U.S. EPA, SCDHEC	12/1/14	1/16/15	NA	NA
Prepare/Submit Draft Final RI Report	Resolution Consultants	1/16/15	5/18/15	Draft Final RI Report	5/18/15
Regulatory Review	U.S. EPA, SCDHEC	5/18/15	7/3/15	NA	NA
Prepare/Submit Final RI Report	Resolution Consultants	7/3/15	7/18/15	Final RI Report	7/18/15

Notes:
SAP = Sampling and Analysis Plan
U.S. EPA = United States Environmental Protection Agency

SCDHEC	=	South Carolina Department of Environmental Control
RI	=	Remedial Investigation
GCAL	=	Gulf Coast Analytical Laboratories
NA	=	Not applicable

SAP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

(UFP-QAPP Manual Section 3.1.1)

17.1 Sampling Approach

This RI/RFI will investigate potential sources of contamination and assess potential threats to human health and the environment, through screening comparisons. The sampling strategy, as presented herein, is designed to address gaps associated with the current dataset and evaluate soil and groundwater for COPCs, including VOCs, SVOCs, pesticides, PCBs, TAL metals, and hexavalent chromium as defined based on historical detections and operations. Furthermore, the proposed strategy considers regulatory comments related to historical documents prepared on behalf of the Site and incorporates input received during partnering sessions, as summarized in Worksheet #9. Findings will provide information to support a decision concerning whether or not future action is required.

This worksheet presents the design and rationale of the sampling and analysis program to be conducted during the RI/RFI. The sampling approach, based on professional judgment, is biased to investigate areas most likely to contain "worst-case" concentrations of COPCs. Data from these "worse-case" areas will facilitate screening efforts necessary to determine whether an impact to human or ecological receptors has occurred. Specifically, the sampling plan was developed using information on historical operations, the preliminary CSM (including subsurface conditions and anticipated groundwater flow directions) as presented in Worksheet #10, assumptions regarding potential contaminant distribution, and data generated during previous investigations. Proposed sampling locations are illustrated on Figures 17-1 and 17-2. A summary table, including sample identification numbers, depth, relevant technical procedures, and applicable laboratory and field analyses, is included as Worksheet #18. Technical procedures for field activities are summarized in Worksheet #21 and included as Appendix A. Professional judgment may be used to adjust sampling locations and/or depths in the field. Examples of criteria, which may lead to changes to the sampling plan, may include visual observations/staining, odors, elevated headspace PID readings, etc.

It should be noted that deviations to the sampling program, including sample location and depth, require review and approval by the Navy, MCRD Parris Island, SCDHEC, and U.S. EPA. The approval process for deviations made in the field is detailed in Worksheet #6.

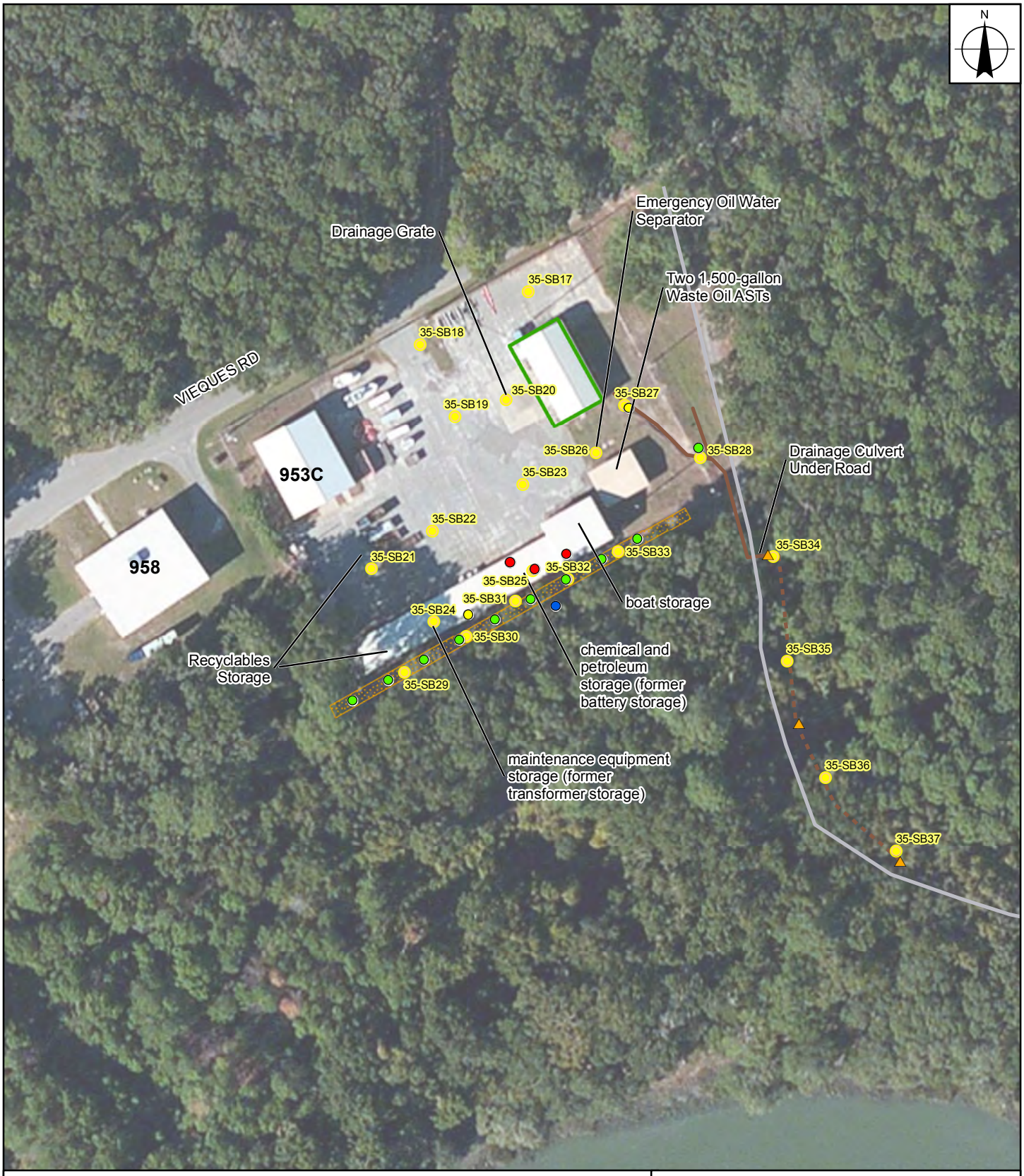
Samples will be submitted to GCAL, a South Carolina-certified laboratory, for chemical analyses. Analytical methods are identified in Worksheet #23. Laboratory SOPs are listed in Worksheet #23 and included in Appendix B. The total numbers of sample analyses to be performed for each target analyte or analytical group are identified in Worksheets #18 and #20. Worksheets #19 and #30 present a summary of the sample analyses, container types and volumes, preservation requirements, and holding times.

Planned field QC samples will include field duplicates and equipment rinsate blanks. Worksheet #12 presents the field QC sample summary. Additional sample volume will be collected as necessary for laboratory QC analysis of matrix spike (MS) and matrix spike duplicate (MSD) samples.

Sample locations will be marked in the field using a wooden stake or brightly colored pin flag. Coordinates of each sample location will be recorded using a GPS device and surveyed for horizontal and vertical location by a South Carolina Professional Land Surveyor, which will allow for future reacquisition of the locations if further investigation or remedial action is necessary. All sample location markers will be removed prior to final demobilization.

17.2 Soil Sampling Program

Up to 42 samples will be collected from 21 soil borings positioned across the Site, as shown of Figure 17-1. Borings will be located at areas historically used for chemical and debris storage prior to the construction of the existing surface cover, beneath cracks at the storage area, and where drainage exits to unpaved surfaces along the southern edge of the Site and the earthen ditch at the southeast corner. Per U.S. EPA request, soil samples in the previously excavated low-lying area at the Site's southern edge, will be collected immediately downgradient of openings where drainage exits the Site's paved area. Soil samples will be collected at the surface (between 0 and 0.5 feet bgs) and immediately above the shallow water table (estimated to occur at less than 10 feet bgs) from each of the 17 soil borings. Soil samples will be submitted to a laboratory for analysis of VOCs by Method 8260B, SVOCs by Method 8270D, polynuclear aromatic hydrocarbons (PAHs) by Method 8270D selective ion monitoring (SIM), pesticides by Method 8081B, PCBs by Method 8082A, TAL metals by Method 6020A/7471A, and hexavalent chromium by Method 7196A.



Proposed Soil Boring/Sample Location

Historical Samples

- 1995 soil sampling location
- 1999 soil sampling location
- 2003 soil sampling location
- 2005 confirmatory soil sampling location
- 1999 groundwater sampling location

Earthen Ditch (dry)

- Ditch Inferred from 2003 Sampling
- Ditch
- 2004 Excavation Area
- 90-day RCRA Hazardous Storage Facility (Building 953)
- Gravel Road

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

1 inch = 100 feet

0 25 50 75 100 Feet

FIGURE 17-1
PROPOSED SOIL SAMPLING LOCATIONS
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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Proposed Monitoring Well

Historical Samples

- 1995 soil sampling location
- 1999 soil sampling location
- 2003 soil sampling location
- 2005 confirmatory soil sampling location
- 1999 groundwater sampling

Earthen Ditch (dry)

- Ditch Inferred from 2003
- Ditch
- 2004 Excavation
- 90-day RCRA Hazardous Storage Facility (Building 953)
- Gravel Road

SOURCE: GOOGLE EARTH PRO
IMAGE DATE: JAN 28, 2011

1 inch = 100 feet

0 25 50 75 100 Feet

FIGURE 17-2
PROPOSED MONITORING WELL LOCATIONS
SITE 35
MCRD PARRIS ISLAND, SOUTH CAROLINA

NAVFAC Naval Facilities Engineering Command
RESOLUTION CONSULTANTS

REQUESTED BY: D. WARREN	DATE: 7/17/2014
DRAWN BY: A. ZIMMERMAN	TASK ORDER NUMBER: JM03

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17.3 Groundwater Sampling Program

As shown on Figure 17-2, the installation, development, and sampling of seven, shallow monitoring wells is planned during this RI/RFI. Monitoring wells will be positioned to evaluate shallow groundwater migrating to the Site from the upgradient direction, at areas within the Site's fenced area, and downgradient of the Site. Well screens will be positioned to intersect the water table. To obtain representative groundwater elevation data for use in evaluating flow direction, wells will be allowed to stabilize prior to gauging depth to water. Synoptic groundwater level measurements will be recorded at the seven wells at least 48 hours following development. Groundwater samples will be collected, through U.S. EPA low-flow sampling protocol, from each of these seven wells and submitted to a laboratory for analysis of VOCs by Method 8260B, SVOCs by Method 8270D, PAHs by Method 8270D SIM, pesticides by Method 8081B, PCBs by Method 8082A, and TAL metals (unfiltered) by Method 6020A/7470A. To confirm that groundwater samples are representative of the formation being investigated, field measurements of water level/drawdown, temperature, pH, specific conductance, oxidation-reduction potential, dissolved oxygen, and turbidity will be recorded.

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SAP WORKSHEET #18: LOCATION-SPECIFIC SAMPLING METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs) [1]	Analytical Group	Number of Samples	Rationale	Sampling Standard Operating Procedure Reference (Worksheet #21/Appendix A)
35-SB17	PI35SB17SO001N1 PI35SB17SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	SESDPROC-005-R2, SESDPROC-011-R4, SESDPROC-012-R3, SESDPROC-205-R2, SESDPROC-209-R2, SESDPROC-300-R2
35-SB18	PI35SB18SO01N1 PI35SB18SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB19	PI35SB19SO01N1 PI35SB19SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB20	PI35SB20SO01N1 PI35SB20SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB21	PI35SB21SO01N1 PI35SB21SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB22	PI35SB22SO01N1 PI35SB22SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB23	PI35SB23SO01N1 PI35SB23SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB24	PI35SB24SO01N1 PI35SB24SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB25	PI35SB25SO01N1 PI35SB25SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB26	PI35SB26SO01N1 PI35SB26SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil within historical storage area.	
35-SB27	PI35SB27SO01N1 PI35SB27SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil at entry point of earthen ditch.	
35-SB28	PI35SB28SO01N1 PI35SB28SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in earthen ditch.	

Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs) [1]	Analytical Group	Number of Samples	Rationale	Sampling Standard Operating Procedure Reference (Worksheet #21/Appendix A)
35-SB29	PI35SB29SO01N1 PI35SB29SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in low-lying area.	
35-SB30	PI35SB30SO01N1 PI35SB30SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in low-lying area.	
35-SB31	PI35SB31SO01N1 PI35SB31SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in low-lying area.	
35-SB32	PI35SB32SO01N1 PI35SB32SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in low-lying area.	
35-SB33	PI35SB33SO01N1 PI35SB33SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in low-lying area.	
35-SB34	PI35SB34SO01N1 PI35SB34SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in earthen ditch.	
35-SB35	PI35SB35SO01N1 PI35SB35SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in earthen ditch.	
35-SB36	PI35SB36SO01N1 PI35SB36SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in earthen ditch.	
35-SB37	PI35SB37SO01N1 PI35SB37SO07N1	Soil	0.0 – 0.5 7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	2	Evaluate surface and subsurface soil in earthen ditch.	
35-SBXX	PI35SBXXSQ01FD1	Soil (duplicate)	0.0 – 0.5	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ01FD2	Soil (duplicate)	0.0 – 0.5	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ01FD3	Soil (duplicate)	0.0 – 0.5	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ07FD4	Soil (duplicate)	7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ07FD5	Soil (duplicate)	7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ07FD6	Soil (duplicate)	7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ01EB1	Soil (Equipment Blank)	NA	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	

Sample Location	Sample Identification	Matrix	Estimated Depth (feet bgs) [1]	Analytical Group	Number of Samples	Rationale	Sampling Standard Operating Procedure Reference (Worksheet #21/Appendix A)
35-SBXX	PI35SBXXSQ01EB2	Soil (Equipment Blank)	NA	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ07EB3	Soil (Equipment Blank)	NA	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSQ07EB4	Soil (Equipment Blank)	NA	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSO01N1 (MS/MSD1)	Soil (MS/MSD1)	0.0 – 0.5	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSO01N1 (MS/MSD2)	Soil (MS/MSD2)	0.0 – 0.5	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSO01N1 (MS/MSD3)	Soil (MS/MSD2)	7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-SBXX	PI35SBXXSO07N1 (MS/MSD4)	Soil (MS/MSD)	7.0 – 7.5 [2]	VOCs, SVOCs, Pesticides, PCBs, TAL Metals, CrVI	1	Quality assurance	
35-MW01	PI35MW01WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater in upgradient direction.	SESDPROC-005-R2, SESDPROC-011-R4, SESDPROC-012-R3, SESDPROC-100-R3, SESDPROC-101-R5, SESDPROC-102-R3, SESDPROC-103-R3, SESDPROC-106-R3, SESDPROC-111-R3, SESDPROC-113-R1, SESDPROC-205-R2, SESDPROC-209-R2, SESDPROC-301-R3
35-MW02	PI35MW02WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater within historical storage area.	
35-MW03	PI35MW03WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater at entry point of earthen ditch.	
35-MW04	PI35MW04WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater downgradient of historical storage area and low-lying area.	
35-MW05	PI35MW05WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater downgradient of historical storage area.	
35-MW06	PI35MW06WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater downgradient of historical storage area and low-lying area.	
35-MW07	PI35MW07WG*MMDDYYR* N1	WG	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Evaluate groundwater downgradient of historical storage area and low-lying area and earthen ditch.	
35-MW0X	PI35MW0XWQ*MMDDYYR* FD1	WG (duplicate)	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Quality assurance	
35-MW0X	PI35MW0XWQ*MMDDYYR* EB1	WG (equipment blank)	NA	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Quality assurance	
35-MW0X	PI35MW0XWG*MMDDYYR* N1 (MS/MSD)	WG (MS/MSD)	Water Table	VOCs, SVOCs, Pesticides, PCBs, TAL Metals	1	Quality assurance	

Notes:

bgs	=	Below ground surface
CrVI	=	Hexavalent chromium
DY	=	Day
SCDHEC	=	South Carolina Department of Health and Environmental Control
YR	=	Year
WG	=	Groundwater
IDW	=	Investigative Derived Waste
VOCs	=	Volatile organic compounds
SVOCs	=	Semi-volatile organic compounds
PCBs	=	Polychlorinated biphenyls
TAL	=	Target Analyte List
NA	=	Not Applicable
MS/MSD	=	Matrix Spike/Matrix Spike Duplicate
XX	=	To Be Determined in Field

[1] Surface soil samples will be collected from native soil at 0.0 to 0.5 feet below ground surface.
[2] Deep sample to be collected approximately 0.5 feet above the shallow water table, assumed to occur at approximately 7.5 ft bgs

SAP WORKSHEET #19: ANALYTICAL METHODS/SOP REQUIREMENTS TABLE

(UFP-QAPP Manual Section 3.1.1)

Laboratory Name and Address: GCAL, 7979 Innovation Park Dr., Baton Rouge, LA 70820
Laboratory Point of Contact/Project Manager: Brenda Martinez, brenda.martinez@gcal.com, (225) 769-4900

Matrix	Analytical Group	Analytical and Preparation Method/ *GCAL SOP Reference*	Containers (number, size, and type)	Sample Volume	Preservation Requirements[1]	Maximum Holding Time[2] (preparation/analysis)
Groundwater	VOCs	SW-846 5030/8260B *GCMSV-003*	(3) 40 mL glass volatile vials	40 mL	Hydrochloric acid to a pH less than 2; Cool to 0-6°C; no headspace	14 days
Soil	VOCs	SW-846 5035/8260B *GCMSV-003*	(3) 40 mL glass plus 2 oz jar	5 grams	Methanol and sodium bisulfate; Cool to 0-6°C	14 days
Groundwater	SVOCs/PAHs via SIM	SW-846 3510C/8270D *EXT-003/GCMSSV-004*	(2) 1 L amber glass bottles	1000 mL	Cool to 0-6°C	7 days to preparation 40 days to analysis
Soil	SVOCs/PAHs via SIM	SW-846 3550C/8270D *EXT-001/GCMSSV-004*	(1) 4 oz glass jar	30 grams	Cool to 0-6°C	14 days to preparation 40 days to analysis
Groundwater	Pesticides	SW-846 3510C/8081B *EXT-010/GC-013*	(2) 1 L amber glass bottles	1000 mL	Cool to 0-6°C	7 days to preparation 40 days to analysis
Soil	Pesticides	SW-846 3550C/8081B *EXT-002/GC-013*	(1) 4 oz glass jar	30 grams	Cool to 0-6°C	14 days to preparation 40 days to analysis
Groundwater	PCBs	SW-846 3510C/8082A *EXT-010/GC-023*	(2) 1 L amber glass bottles	1000 mL	Cool to 0-6°C	7 days to preparation 40 days to analysis
Soil	PCBs	SW-846 3550C/8082A *EXT-002/GC-023*	(1) 4 oz glass jar	30 grams	Cool to 0-6°C	14 days to preparation 40 days to analysis
Groundwater	Metals	SW-846 3010A/6020A *MET-005/MET-021*	(1) 250-mL plastic bottle	50 mL	Nitric acid to a pH less than 2	180 days
Groundwater	Mercury	SW-846 7470A *MET-006/MET-008*		20 mL	Nitric acid to a pH less than 2	28 days

Laboratory Name and Address: GCAL, 7979 Innovation Park Dr., Baton Rouge, LA 70820
Laboratory Point of Contact/Project Manager: Brenda Martinez, brenda.martinez@gcal.com, (225) 769-4900

Matrix	Analytical Group	Analytical and Preparation Method/ *GCAL SOP Reference*	Containers (number, size, and type)	Sample Volume	Preservation Requirements(1)	Maximum Holding Time(2) (preparation/analysis)
Soil	Metals	SW-846 3050B/6020A *MET-004/MET-021*	(1) 4 oz glass jar	1.25 grams	None	180 days
Soil	Mercury	SW-846 7471B *MET-006/MET-008*		0.6 grams	Cool to 0-6°C	28 days
Soil	Hexavalent Chromium	SW-846 3060A/7196A *WL-012*		5 grams	Cool 0-6°C	30 days to preparation 7 days to analysis
Water (Equipment Blank)	Hexavalent Chromium	SW-846 7196A *WL-012*	(1) 250-mL plastic bottle	100 mL	Cool to 0-6°C	24 hours

Notes:

(1) = Chemical, temperature, and light protected
(2) = Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted
GCAL = Gulf Coast Analytical Laboratory
SOP = Standard operating procedure
VOCs = Volatile organic compounds
SVOCs = Semi-volatile organic compounds
PAHs = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
PCBs = Polychlorinated biphenyls
mL = Milliliter
°C = Degrees Celsius
oz = Ounce
L = Liter

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(1)	No. of Equipment Blanks	Total No. of Samples to Lab
Surface Soil	VOCs	21	3	2	2	28
	SVOCs	21	3	2	2	28
	Pesticides	21	3	2	2	28
	PCBs	21	3	2	2	28
	Metals	21	3	2	2	28
Subsurface Soil	VOCs	21	3	2	2	28
	SVOCs	21	3	2	2	28
	Pesticides	21	3	2	2	28
	PCBs	21	3	2	2	28
	Metals	21	3	2	2	28
Groundwater	VOCs	7	1	1	1	10
	SVOCs	7	1	1	1	10
	Pesticides	7	1	1	1	10
	PCBs	7	1	1	1	10
	Metals	7	1	1	1	10

Notes:

[1] Although MS/MSD samples are not typically considered field QC samples, they are included here because location determination is often established in the field. MS/MSD samples are not included in the total number of samples sent to the laboratory. For total metals, an MD will be collected in place of an MSD.

VOCs = Volatile organic compounds
SVOCs = Semi-volatile organic compounds
PCBs = Polychlorinated biphenyls
MS = Matrix Spike
MSD = Matrix Spike Duplicate

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SAP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.1.2)

Field SOPs Reference Table					
SOP Reference Number	**Title/Author**	**Revision Date or Version Number**	**Location of SOP**	**Any planned deviation for Project Work**	**Comments**
R.61-71	*SCDHEC Well Standards,* South Carolina Department of Health and Environmental Control	26 April 2002	Appendix A	No	
SESDGUID-101-R1	*Design and Installation of Monitoring Wells,* United States Environmental Protection Agency, Region 4	29 January 2013	Appendix A	No	
SESDPROC-005-R2	*Sample and Evidence Management,* United States Environmental Protection Agency, Region 4	29 January 2013	Appendix A	No	
SESDPROC-010-R5	*Logbooks,* United States Environmental Protection Agency, Region 4	30 May 2013	Appendix A	No	
SESDPROC-011-R4	*Field Sampling Quality Control*, United States Environmental Protection Agency, Region 4	5 February 2013	Appendix A	No	
SESDPROC-012-R3	*Field Sampling and Measurement Procedures and Procedure Validation*, United States Environmental Protection Agency, Region 4	30 August 2012	Appendix A	No	
SESDPROC-100-R3	*Field pH Measurement*, United States Environmental Protection Agency, Region 4	29 January 2013	Appendix A	No	
SESDPROC-101-R5	*Field Specific Conductance Measurement*, United States Environmental Protection Agency, Region 4	30 August 2012	Appendix A	No	
SESDPROC-102-R3	*Field Temperature Measurement*, United States Environmental Protection Agency, Region 4	4 February 2011	Appendix A	No	
SESDPROC-103-R3	*Field Turbidity Measurement, United States Environmental Protection Agency, Region* 4	29 January 2013	Appendix A	No	
SESDPROC-105-R2	*Groundwater Level and Well Depth Measurement*, United States Environmental Protection Agency, Region 4	29 January 2013	Appendix A	No	
SESDPROC-106-R3	*Field Measurement of Dissolved Oxygen*, United States Environmental Protection Agency, Region 4	8 January 2014	Appendix A	No	
SESDPROC-108-R3	*Equipment Inventory and Management*, United States Environmental Protection Agency, Region 4	6 February 2013	Appendix A	No	

Field SOPs Reference Table					
SOP Reference Number	**Title/Author**	**Revision Date or Version Number**	**Location of SOP**	**Any planned deviation for Project Work**	**Comments**
SESDPROC-110-R3	*Global Positioning System*, United States Environmental Protection Agency, Region 4	20 April 2011	Appendix A	No	
SESDPROC-111-R3	*In-Situ Water Quality Monitoring*, United States Environmental Protection Agency, Region 4	19 July 2013	Appendix A	No	
SESDPROC-113-R1	*Field Measurement of ORP*, United States Environmental Protection Agency, Region 4	29 January 2013	Appendix A	No	
SESDPROC-202-R2	*Management of Investigation Derived Waste,* United States Environmental Protection Agency, Region 4	15 October 2010	Appendix A	No	
SESDPROC-205-R2	*Field Equipment Cleaning and Decontamination*, United States Environmental Protection Agency, Region 4	20 December 2011	Appendix A	No	
SESDPROC-209-R2	*Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples*, United States Environmental Protection Agency, Region 4	20 April 2011	Appendix A	No	
SESDPROC-300-R2	*Soil Sampling*, United States Environmental Protection Agency, Region 4	20 December 2011	Appendix A	No	
SESDPROC-301-R3	*Groundwater Sampling*, United States Environmental Protection Agency, Region 4	6 March 2013	Appendix A	No	

SAP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criterion	Corrective Action	Responsible Person	SOP Reference[1]	Comments
YSI 600 Series Water Quality Meter	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	To be used to determine purge completion
Turbidity Meter (LaMotte 2020 or equivalent)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance Calibrations must bracket expected values. Initial Calibration Verification must be <5 Nephelometric Turbidity Unit.	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	To be used to determine purge completion
Photoionization Detector	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-20	To be used to assist with determination of sampling depths and for safety monitoring
Water Level Indicator and Oil/Water Interface Probe	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP-3-24	None
Global Positioning System	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision < 3, number of satellites must be at least six	Wait for better signal, replace unit, or choose alternate location technique	Resolution Consultants FOL or designee	Manufacturer's Guidance Manual, SOP SESDPROC-110-R3	None

Notes:

[1] = Specify the appropriate reference letter or number from the Project Sampling SOP References Table (Worksheet #21)
FOL = Field operations leader
SOP = Standard operating procedure

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SAP WORKSHEET #23: ANALYTICAL SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.2.1)

Laboratory Name and Address: GCAL, 7979 Innovation Park Dr., Baton Rouge, LA 70820
Laboratory Point of Contact/Project Manager: Brenda Martinez, brenda.martinez@gcal.com, (225) 769-4900

GCAL SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
GCMSV-003	*Standard Operating Procedure for Analysis of Volatile Organic Compounds by GC/MS, SW-846 8260B*, Revision 24, 10/3/13	Definitive	Aqueous and Soil, VOC	GC/MS Agilent 6890/5973 and Agilent 7890/5975	No	N
GCMSSV-004	*Standard Operating Procedure for Analysis of Semi-Volatile Organic Compounds by GC/MS, EPA 8270D*, Revision 9, 3/14/14	Definitive	Aqueous and Soil, SVOC, PAHs via SIM	GC/MS Agilent 6890/5973 and Agilent 7890/5975	No	N
EXT-003	*Standard Operating Procedure for Separatory Funnel Liquid/Liquid Extraction for Base/Neutral and Acid Compounds*, Revision 24, 3/26/14	Definitive	Aqueous, SVOC and PAHs via SIM Extraction	NA	No	N
EXT-001	*Standard Operating Procedure for Ultrasonic Extraction for Base/Neutral and Acid Compounds,* Revision 20, 4/11/14	Definitive	Soil, SVOC and PAHs via SIM Extraction	NA	No	N
GC-012	*Standard Operating Procedure for Organchlorine Pesticides and PCBs by Gas Chromatography, EPA 608/EPA8081B/EPA8082A,* Revision 10, 4/11/14	Definitive	Aqueous and Soil, Pesticides	Agilent 6890N GC/ECD	No	N
EXT-010	*Standard Operating Procedure for Separatory Funnel Liquid/Liquid Extraction for Pesticide and PCBs, SW-846 3050C, EPA 608*, Revision 16, 1/10/14	Definitive	Aqueous, Pesticide/PCB Extraction	NA	No	N
EXT-002	*Standard Operating Procedure for Ultrasonic Extraction for Pesticides and PCBs*, Revision 20, 4/4/14	Definitive	Soil, Pesticide/PCB Extraction	NA	No	N
MET-021	*Standard Operating Procedure for Metals Analysis by Inductively Coupled Plasma-Mass Spectrometry, EPA 200.8/EPA 6020A,* Revision 5, 3/7/14	Definitive	Aqueous and Soil, Metals	Agilent 7700	No	N
MET-005	*Standard Operating Procedure for Acid Digestion of Aqueous Samples for Metals Analysis, SW-846 3010A,* Revision 16, 10/8/13	Definitive	Aqueous, ICP Metals Digestion	NA	No	N

Laboratory Name and Address: GCAL, 7979 Innovation Park Dr., Baton Rouge, LA 70820
Laboratory Point of Contact/Project Manager: Brenda Martinez, brenda.martinez@gcal.com, (225) 769-4900

GCAL SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM	Modified for Project Work? (Y/N)
MET-004	*Standard Operating Procedure for Acid Digestion of Solids Samples for Metals Analysis, SW-846 3050B*, Revision 16, 10/8/13	Definitive	Soil, ICP Metals Digestion	NA	No	N
MET-008	*Standard Operating Procedure for Analysis of Aqueous and Solid Samples for Mercury, EPA 245.2/EPA 7470A/EPA7471B*, Revision 19, 12/5/13	Definitive	Aqueous and Soil, Mercury	Perkin Elmer FIMS 400	No	N
MET-006	*Standard Operating Procedure for Acid Digestion of Aqueous and Solid Samples for Mercury Analysis, EPA 245.2/EPA 7470A/EPA 7471B,* Revision 20, 12/5/13	Definitive	Aqueous and Soil, Mercury Digestion	NA	No	N
WL-012	*Standard Operating Procedure for Hexavalent Chromium,* Revision 14, 5/16/14	Definitive	Soil and Water Metals	HACH DR2800 Spectrophotometer	No	Y [1]

Notes:
[1] Soil sample weight will be changed from 2.5 grams to 5 grams to obtain lower detection limits.
GCAL = Gulf Coast Analytical Laboratory
SOP = Standard operating procedure
QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010 (and subsequent revisions)
Y/N = Yes/No
GC/MS = Gas chromatograph/mass spectrometer
VOC = Volatile organic compounds
SVOC = Semi-volatile organic compounds
PAH = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
GC/ECD = Gas chromatography/electron capture detector
PCB = Polychlorinated biphenyl compounds

SAP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION TABLE

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
GCMS VOCs	CCV	CV daily, before sample analysis, and every 12 hours of analysis time	All targets ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	GCAL SOP GCMSV-003
GCMS VOCs	ICAL Minimum five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD ≤ 15% for all compounds, linear or quadratic curve fit with COD ≥ 0.99	Repeat calibration if criterion is not met	Analyst, Supervisor	GCAL SOP GCMSV-003
GCMS VOCs	Second source CV	Once after each ICAL	All analytes within ± 20% of expected value	Remake standard, recalibrate if necessary	Analyst, Supervisor	GCAL SOP GCMSV-003
GCMS VOCs	RRT Evaluation	Prior to sample analysis	Set at mid-point of ICAL; +/- 30 seconds each CCV	CCV fails, perform column maintenance, inspect pumps, and leak checks	Analyst, Supervisor	GCAL SOP GCMSV-003
GCMS SVOCs/PAHs via SIM	Breakdown check (DDT)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem then repeat the breakdown check. No samples shall be run until degradation ≤ 20%.	Analyst, Supervisor	GCAL SOP GCMSSV-004
GCMS SVOCs/PAHs via SIM	CCV	CV daily, before sample analysis, and every 12 hours of analysis time	All targets ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	GCAL SOP GCMSSV-004
GCMS SVOCs/PAHs via SIM	ICAL Minimum five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD ≤ 15% for all compounds, linear or quadratic curve fit with COD ≥ 0.99	Repeat calibration if criterion is not met	Analyst, Supervisor	GCAL SOP GCMSSV-004
GCMS SVOCs/PAHs via SIM	Second source CV	Once after each ICAL	All analytes within ± 20% of expected value	Remake standard, recalibrate if necessary	Analyst, Supervisor	GCAL SOP GCMSSV-004
GCMS SVOCs/PAHs via SIM	RRT Evaluation	Prior to sample analysis	Set at mid-point of ICAL; +/- 30 seconds each CCV	CCV fails, perform column maintenance, inspect pumps, and leak checks	Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/ECD Pesticides	Breakdown check (Endrin/DDT Method 8081B)	Each day prior to analysis and every 12 hours.	Breakdown of Endrin and DDT not to exceed 15%	Instrument maintenance. Sample analysis cannot proceed until breakdown criteria are achieved.	Analyst, Supervisor	GCAL SOP GC-012

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
GC/ECD Pesticides	ICAL Minimum five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD <20% for all compounds or linear or quadratic calibration r2 > 0.990	Repeat calibration if criterion is not met	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	Second source CV	Once after each ICAL	All analytes within ± 15% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	ICV and CCV	CCV after every 10 samples and at the end of the analytical sequence	%D or Drift < 20% for all analytes	Repeat initial calibration and reanalyze all samples analyzed since the last successful Calibration verification.	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	Confirmation of positive results (second column or second detector)	All samples and QC	Detections agree within 40%	Report the result from the primary column and include a narrative unless matrix interference is creating a high bias.	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	RT window width calculated for each analyte and surrogate	After installing a new column, performing major maintenance or at initial set-up	Width is 3 times standard deviation of a minimum of three injections over a minimum of 72 hours, or default of 0.03 minutes whichever is greater.	Not applicable	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	RT Window position establishment for each analyte and surrogate	Set using mid-point of ICAL or at first CCV of the day if ICAL is not performed.	Not applicable	Not applicable	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD Pesticides	Retention time window verification for each analyte and surrogate	Set using mid-point of ICAL or at first CCV of the day if ICAL is not performed.	Not applicable	Not applicable	Analyst, Supervisor	GCAL SOP GC-12
GC/ECD PCBs	ICAL Minimum five-point for Aroclor 1016/1260, one point calibration of remaining Aroclors	Initial calibration prior to sample analysis	RSD ≤20% for all compounds or linear or quadratic calibration r2 > 0.990	Repeat calibration if criterion is not met.	Analyst, Supervisor	GCAL SOP GC-12
GC/ECD PCBs	Second source calibration verification	Once after each initial calibration	All analytes within ± 15% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP GC-012

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
GC/ECD PCBs	ICV and CCV	CCV after every 10 samples and at the end of the analytical sequence	%D or Drift < 20% for all analytes	Repeat initial calibration and reanalyze all samples analyzed since the last successful Calibration verification.	Analyst, Supervisor	GCAL SOP GC-012
GC/ECD PCBs	Confirmation of positive results (second column or second detector)	All samples and QC	Detections agree within 40%	Report the result from the primary column and include a narrative unless matrix interference is creating a high bias.	Analyst, Supervisor	GCAL SOP GC-012
ICP/MS Metals	ICAL for all analytes: minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, r2 ≥0.995	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Metals	Second source ICV	Once after each initial calibration	All analytes within ± 10% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Metals	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within ± 10% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Metals	Low-level calibration check standard	Daily following calibration	80-120% recovery	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Metals	Calibration blank	Once with each ICAL, after every 10 samples and at the end of an analytical sequence.	Determined concentration ≤ LOD	Determine source of possible contamination, perform maintenance and recalibrate.	Analyst, Supervisor	GCAL SOP MET-021
ICP/MS Metals	Linear Dynamic range/High level Check	Every 6 months and with major maintenance	90-110% recovery	Perform maintenance and/or reanalyze at lower concentration.	Analyst, Supervisor	GCAL SOP MET-021
CVAA Mercury	ICAL — Minimum 5 standards and blank	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.9995	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-008
CVAA Mercury	ICV	Once after each initial calibration	All analytes within ± 10% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-008

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	GCAL SOP Reference
CVAA Mercury	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within ± 10% of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Analyst, Supervisor	GCAL SOP MET-008
CVAA Mercury	Calibration Blank	Once with each ICAL, after every 10 samples and at the end of an analytical sequence.	Determined concentration ≤ LOD	Determine source of possible contamination, perform maintenance and recalibrate.	Analyst, Supervisor	GCAL SOP MET-008
Spectro-photometer Cr VI	ICAL – Minimum of five standards and a blank	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995	Recalibrate and/or perform necessary equipment maintenance, remake standards or replace reagents as appropriate.	Analyst, Supervisor	GCAL SOP WL-012
Spectro-photometer Cr VI	CCV	CCV to bracket every ten samples and at the end of an analytical sequence	Recovery within ± 10% of expected value	Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification.	Analyst, Supervisor	GCAL SOP WL-012
Spectro-photometer Cr VI	ICV	Once after an initial calibration	Recovery within ± 10% of expected value	Recalibrate using fresh standards and/or reagents as appropriate. Perform maintenance if required. Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat calibration.	Analyst, Supervisor	GCAL SOP WL-012
Spectro-photometer Cr VI	Calibration Blank	Once with each ICAL, after every 10 samples, and at the end of an analytical sequence	Determined concentration ≤ LOD	Determine source of possible contamination, perform maintenance as required and re-analyze affected samples. Evaluate ICAL to determine if issue is an artifact of curve fit, recalibrate if required.	Analyst, Supervisor	GCAL SOP WL-012

Notes:

GCAL = Gulf Coast Analytical Laboratory
SOP = Standard operating procedure
GCMS = Gas chromatograph/mass spectrometer
VOCs = Volatile organic compounds
SVOCs = Semi-volatile organic compounds
PAH = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
PCBs = Polychlorinated biphenyl compounds

CCV	=	Continuing calibration verification
CV	=	Calibration verification
%D	=	Percent difference
ICAL	=	Initial calibration
%RSD	=	Relative standard deviation
RFs	=	Response factors
CCCs	=	Calibration check compounds
COD	=	Coefficient of the determination
RRT	=	Relative retention times
GC/ECD	=	Gas chromatography/electron capture detector
DDT	=	4,4'-Dichlorodiphenyltrichloroethane
QC	=	Quality control
r2	=	Correlation coefficient
ICV	=	Initial calibration verification
RT	=	Retention time
ICP/MS	=	Inductively coupled plasma spectroscopy/mass spectrometer
CVAA	=	Cold vapor atomic absorption
LOD	=	Limit of detection
CR VI	=	Hexavalent chromium

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SAP WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	GCAL SOP Reference
GC/MS	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner	VOCs	Monitor instrument performance via Continuing Calibration Verification	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Replace connections, clean source, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Analyst, Supervisor	GCAL SOP GCMSV-003
GC/MS	Check for leaks, replace gas line filters, replace column, clean injection port/liner	SVOCs/ PAHs via SIM	Monitor instrument performance via Continuing Calibration Verification	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Replace connections, clean source, replace gas line filters, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Analyst, Supervisor	GCAL SOP GCMSSV-004
GC/ECD	Clean injection port and replace liner Clip column Maintain pumps ECD wipe test	Pesticides	Monitor instrument performance via Continuing Calibration	Daily Wipe test annually	Calibration and QC criteria met	Change column Instrument maintenance	Analyst, Supervisor	GCAL SOP GC-013
GC/ECD	Clean injection port and replace liner Clip column Maintain pumps ECD wipe test	PCBs	Monitor instrument performance via Continuing Calibration	Daily Wipe test annually	Calibration and QC criteria met	Change column Instrument maintenance	Analyst, Supervisor	GCAL SOP GC-023
ICP/MS	Change pump tubing, clean nebulizer, change torch, clean sample cone/skimmer cone	Metals	Monitor instrument performance via Continuing Calibration Verification and CCBlank	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Change pump tubing, change torch and nebulizer, clean cone; recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP MET-021
CVAA	Check pump tubing, change sample tubing at least daily. Change reductant, carrier and waste tubing	Mercury	Monitor instrument performance via Continuing Calibration Verification and CCBlank	As needed	No maintenance is required as long as instrument QC meets DoD QSM criteria	Change pump tubing, recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP MET-008

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	GCAL SOP Reference
Spectro-photometer	Inspect Cell Lamp Maintenance	Cr VI	Monitior instrument performance via CCV and Blank Performance of wavelength and photo verification standard	Inspect cell as needed; standard analyzed quarterly	Cell maintenance is not required as long as instrument QC meets DoD QSM criteria; Wavelength verification ± 2nm and 3%	Clean or replace as necessary, call for service as required	Analyst, Supervisor, QS	GCAL SOP WL-012

Notes:

GCAL = Gulf Coast Analytical Laboratory
SOP = Standard operating procedure
GC/MS = Gas chromatograph/mass spectrometer
VOCs = Volatile organic compounds
SVOCs = Semi-volatile organic compounds
PAHs = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
PCBs = Polychlorinated biphenyl compounds
QC = Quality control
DoD = Department of Defense
QSM = Quality Systems Manual
GC = Gas chromatography
GC/ECD = Gas chromatography/electron capture detector
ICP/MS = Inductively coupled plasma spectroscopy/mass spectrometer
CVAA = Cold vapor atomic absorption
Cr VI = Hexavalent chromium

SAP WORKSHEET #26: SAMPLE HANDLING SYSTEM

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization):	FOL/Resolution Consultants
Sample Packaging (Personnel/Organization):	FOL/Resolution Consultants
Coordination of Shipment (Personnel/Organization):	FOL/Resolution Consultants
Type of Shipment/Carrier:	Overnight via FedEx
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization):	Sample Receiving Supervisor, Charlotte Saucier/GCAL
Sample Custody and Storage (Personnel/Organization):	Sample Receiving Supervisor, Charlotte Saucier/GCAL
Sample Preparation (Personnel/Organization):	Extractions, Prep Supervisor, Rob Martin/GCAL
Sample Determinative Analysis (Personnel/Organization):	Laboratory Manager, Scott Bailey/GCAL
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection):	180 Days from Receipt of Samples
Sample Extract/Digestate Storage (No. of days from extraction/digestion):	180 Days from Receipt of Samples
SAMPLE DISPOSAL	
Personnel/Organization:	Waste Compliance Manager John Bailey/GCAL
Number of Days from Analysis:	180 Days from Receipt of Samples

Notes:
FOL = Field Operations Leader
GCAL = Gulf Coast Analytical Laboratories

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SAP WORKSHEET #27: SAMPLE CUSTODY REQUIREMENTS

(UFP-QAPP Manual Section 3.3.3)

27.1 Sample Nomenclature, Sample Collection Documentation, Handling, and Tracking Procedures

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

27.1.1 Sample Nomenclature

Sample labeling will be conducted in general accordance with the procedures outlined in Worksheet 18. Nomenclature for solid (soil) samples includes the site being investigated, soil boring identification number, sample media, sample depth, and sample type code (i.e., normal, field duplicate, equipment blank, etc.). Nomenclature for aqueous samples includes the site being investigated, monitoring well identification number, sample media, sample date, and sample type code (i.e., normal, field duplicate, equipment blank, etc.). Sample type codes planned for this event will include N for normal samples, FD for field duplicates, and EB for equipment blanks. Field trip blanks will be labeled sequentially followed by the date (i.e., TB-20131213,). Samples to be used for matrix spike (MS) and matrix spike duplicate (MSD) will be labeled MS/MSD on the container label and noted on the chain-of-custody; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Worksheet #18 provides anticipated sample identifiers for this scope of work.

27.1.2 Sample Collection Documentation

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

Field sample log sheets will be used to document sample collection details and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration.

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 involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, arrival and departure times
- Health and safety issues, including personal protective equipment

27.1.3 Sample Handling and Tracking System

Following collection, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 0 to 6 degrees Celsius (°C) until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., FedEx). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Further details on chain-of-custody procedures are provided in SESDPROC-209-R2: *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples* and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A.

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

Resolution Consultants personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Resolution Consultants' personnel and placed into sealed coolers under chain-of-custody in accordance with SESDPROC-209-R2: *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples* and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.

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

27.2 Field Sample Custody Procedures

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with SESDPROC-209-R2: *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples* and SESDPROC-005-R2: *Sample and Evidence Management*, included in Appendix A.

A sample is under custody if:

- The sample is in the physical possession of an authorized person
- The sample is in view of an authorized person after being in his/her possession

- The sample is placed in a secure area by an authorized person after being in his/her possession

- The sample is in a secure area, restricted to authorized personnel only

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs. Integrity of the samples collected will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

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

27.3 Laboratory Chain-of-Custody

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

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	VOCs
Analytical Method	SW-846 8260B
SOP Reference	GCAL SOP GCMSV-003

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect MS or GC for malfunctions. Reanalyze all samples with internal standard failures. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds; EICP area within -50% to +100% of midpoint of ICAL
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

Notes:

VOCs = Volatile organic compounds
SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
DoD QSM = Department of Defense Quality Systems Manual
LCS = Laboratory control sample
ICAL = Initial calibration
EICP = Extracted ion current profile
MS = Mass Spectrometer
GC = Gas chromatograph
MS/MSD = Matrix spike/matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	SVOCs/PAHs via SIM
Analytical Method	SW-846 8270D
SOP Reference	GCAL SOP GCMSSV-004

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard	Inspect MS or GC for malfunctions. Reanalyze all samples with internal standard. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds; EICP area within -50% to +100% of midpoint of ICAL
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

Notes:

SVOCs = Semi-volatile organic compounds
PAHs = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
DoD QSM = Department of Defense Quality Systems Manual
LCS = Laboratory control sample
ICAL = Initial calibration
EICP = Extracted ion current profile
MS = Mass Spectrometer
GC = Gas chromatograph
MS/MSD = Matrix spike/matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	Pesticides
Analytical Method	SW-846 8081B
SOP Reference	GCAL SOP GC-013

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; Re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

Notes:

SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
DoD QSM = Department of Defense Quality Systems Manual
LCS = Laboratory control sample
MS/MSD = Matrix spike/matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	PCBs
Analytical Method	SW-846 8082A
SOP Reference	GCAL SOP GC-023

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
Surrogates	All field and QC samples	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze if sufficient sample is available. If reanalysis confirms failing recoveries, report and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
MS/MSD	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	For matrix evaluation, use LCS recovery criteria; RPD ≤30%.

Notes:

PCBs = Polychlorinated biphenyl compounds
SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
DoD QSM = Department of Defense Quality Systems Manual
LCS = Laboratory control sample
MS/MSD = Matrix spike/matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	Metals
Analytical Method	SW-846 6020A
SOP Reference	GCAL SOP MET-021

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DoD QSM Version 4.2
Internal Standards	In all field samples and standards	Internal standard intensity within 30-120% of intensity of the IS in the ICAL.	Reanalyze all samples with internal standard failures. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Internal standard intensity within 30-120% of intensity of the IS in the ICAL.
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	For matrix evaluation, use LCS recovery criteria

Matrix	Groundwater, Soil
Analytical Group	Metals
Analytical Method	SW-846 6020A
SOP Reference	GCAL SOP MET-021

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Dilution Test	One per preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 x LOQ	Perform Post Digestion Spike	Analyst, Supervisor, QA Manager	Accuracy Bias	Five-fold dilution must agree within ± 10% of the original measurement for samples with concentrations > 50 times LOQ
Post Digestion Spike	When dilution test fails or analyte concentration in all samples < 50 x LOD	Recovery 75-125%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 75-125%

Notes:

SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
LCS = Laboratory control sample
DoD QSM = Department of Defense Quality Systems Manual
ICAL = Initial calibration
MSD = Matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater, Soil
Analytical Group	Mercury
Analytical Method	SW-846 7470A/7471B
SOP Reference	GCAL SOP MET-008

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	QC acceptance criteria specified in DoD QSM Version 4.2	Re-prepare and/or reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DOD QSM Version 4.2
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	For matrix evaluation, use LCS recovery criteria	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	For matrix evaluation, use LCS recovery criteria

Notes:

SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
IS = Internal Standard
LCS = Laboratory control sample
DoD QSM = Department of Defense Quality Systems Manual
ICAL = Initial calibration
MSD = Matrix spike duplicate
RPD = Relative percent difference

SAP WORKSHEET #28: LABORATORY QC SAMPLES TABLE (CONTINUED)

(UFP-QAPP Manual Section 3.4)

Matrix	Soil
Analytical Group	Chromium VI
Analytical Method	SW-846 3060A/7196A
SOP Reference	GCAL SOP WL-012

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Method Blank	One per preparatory batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).	Correct problem; re-prepare and/or reanalyze any sample associated with a blank that fails criteria.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > LOQ (see Box D-1 in QSM V4.2).
LCS	One LCS per preparatory batch	80-120% recovery	Re-prepare and reanalyze all associated samples.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria specified in DOD QSM Version 4.2
Sample Duplicate or MSD	One per preparatory batch	RPD ≤ 20%	Contact the client to determine if additional measures are required. Evaluate matrix to determine homogeneity of sample.	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
Matrix Spike	One per preparatory batch per matrix	75-125% recovery	Evaluate both soluble and insoluble matrix spike results, post-digestion spike recoveries, ORP, and pH data. Failing matrix spike with a reducing environment of the native sample indicates the unlikelihood of the existence of Cr VI in the native sample. Contact client for further instructions.	Analyst, Supervisor, QA Manager	Accuracy Bias	75-125% recovery
Soluble and Insoluble Pre-digestion Matrix	One per preparatory batch per matrix	75-125% recovery	Evaluate both soluble and insoluble matrix spike results, post-digestion spike recoveries, ORP, and pH data.	Analyst, Supervisor, QA Manager	Accuracy Bias	75-125% recovery

Matrix	Soil
Analytical Group	Chromium VI
Analytical Method	SW-846 3060A/7196A
SOP Reference	GCAL SOP WL-012

QC Sample	Frequency/ Number	Method/SOP — QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicators	Measurement Performance Criteria
Spike			Failing matrix spike with a reducing environment of the native sample indicates the unlikelihood of the existence of Cr VI in the native sample. Contact client for further instructions.			
Post Digestion Spike	One per preparatory batch per matrix	85-115% recovery	Evaluate both soluble and insoluble matrix spike results, post-digestion spike recoveries, ORP, and pH data. Failing matrix spike with a reducing environment of the native sample indicates the unlikelihood of the existence of Cr VI in the native sample. Contact client for further instructions.	Analyst, Supervisor, QA Manager	Accuracy Bias	85-115% recovery
ORP and PH determinations	Each sample	Not applicable	Plot ORP and pH data on phase diagram to assess whether matrix is oxidizing or reducing and provide data to client.	Analyst, Supervisor, QA Manager	Accuracy Bias	Determines reduction/oxidation potential of the soil to assist in the evaluation of matrix interferences.

Notes:

SOP = Standard operating procedure
GCAL = Gulf Coast Analytical
QC = Quality assurance
LOQ = Limit of quantitation
QA = Quality assurance
IS = Internal Standard
LCS = Laboratory control sample
DoD QSM = Department of Defense Quality Systems Manual
ICAL = Initial calibration
MSD = Matrix spike duplicate
RPD = Relative percent difference
CR VI = Hexavalent chromium
ORP = Oxidation reduction potential

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SAP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records Project personnel sign-off record Field logbook (and sampling notes) Field sample forms (e.g., sample log sheets, drilling logs, etc.) Chain-of-custody records Sample shipment air bills Equipment calibration logs Photographs Sampling and Analysis Plan Field sampling standard operating procedures Safe work permit forms	Resolution Consultants project file (may include hard copy as well as electronic information).
Analytical Results Documents and Records Sample receipt/log-in forms Sample preparation logs Equipment calibration logs Sample analysis run logs Reported field sample results and raw data Reported results for standards, quality control checks Reported results for standards, quality control samples Data completeness checklists Data validation memoranda	Resolution Consultants project file (may include hard-copy as well as electronic information), long-term data package storage at third-party secure professional document storage firm. Electronic analytical results will be maintained in a database on a password protected Structured Query Language server.
Other Documents Health and Safety Plan All versions of Sampling and Analysis Plan All versions of reports (e.g., status reports, investigation report, etc.)	All versions of the subject document and all support documents will be stored in hard-copy in the Resolution Consultants project file and electronically in the server library.
Final Document/Records Repository Administrative Record files (e.g., progress reports, validation reports, investigation report) Site files Post decision Files Analytical data Spatial data Maps	All final documents and records will be stored in accordance with in the Navy's Environmental Restoration Recordkeeping Manual. Resolution Consultants will update and manage the project related documents, data and maps in the Navy's Naval Installation Restoration Information Solution data management system.

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SAP WORKSHEET #30: ANALYTICAL SERVICES TABLE

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method/SOP	Data Package TAT	Laboratory/Organization[1] (name and address, contact person and telephone number)	Backup Laboratory
Groundwater	VOCs	See Worksheet #18	SW-846 5030/8260B GCMSV-003	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	VOCs	See Worksheet #18	SW-846 5035/8260B GCMSV-003	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Groundwater	SVOCs/PAHs via SIM	See Worksheet #18	SW-846 3510C/8270D EXT-003/GCMSSV-004	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	SVOCs/PAHs via SIM	See Worksheet #18	SW-846 3550C/8270D EXT-001/GCMSSV-004	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Groundwater	Pesticides	See Worksheet #18	SW-846 3510C/8081B EXT-010/GC-013	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	Pesticides	See Worksheet #18	SW-846 3550C/8081B EXT-002/GC-013	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Groundwater	PCBs	See Worksheet #18	SW-846 3510C/8082A EXT-010/GC-023	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	PCBs	See Worksheet #18	SW-846 3550C/8082A EXT-002/GC-023	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Groundwater	Metals	See Worksheet #18	SW-846 3010A/6020A MET-005/MET-021	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Groundwater	Mercury	See Worksheet #18	SW-846 7470A MET-006/MET-008	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	Metals	See Worksheet #18	SW-846 3050B/6020A MET-004/MET-021	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	Mercury	See Worksheet #18	SW-846 7471B MET-006/MET-008	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None
Soil	Hexavalent Chromium	See Worksheet #18	SW-846 3060A/7196A WL-012	21 Days	GCAL, 7979 GSRI Road, Baton Rouge, Louisiana 70820 Brenda Martinez, 225-769-4900	None

Notes:

[1] Laboratory meets accreditation requirements to support project needs.

VOC = Volatile organic compounds
SVOC = Semi-volatile organic compounds
PAH = Polynuclear aromatic compounds
SIM = Selective ion monitoring
PCBs = Polychlorinated biphenyl compounds
GCAL = Gulf Coast Analytical

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SAP WORKSHEET #31: PLANNED PROJECT ASSESSMENTS TABLE

(UFP-QAPP Manual Section 4.1.1)

Worksheet Not Applicable

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SAP WORKSHEET #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES TABLE

(UFP-QAPP Manual Section 4.1.2)

Worksheet Not Applicable

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SAP WORKSHEET #33: QUALITY ASSURANCE MANAGEMENT REPORTS TABLE

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group	Within 4 weeks after receiving the data from the laboratory	Project Chemist or Data Validator, Resolution Consultants	TOM, Resolution Consultants; project file
Project Monthly Progress Report	Monthly for duration of the project	Monthly	TOM, Resolution Consultants	TOM, Resolution Consultants; Program Manager, Resolution Consultants; NAVFAC SE RPM; project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, GCAL	TOM and project file, Resolution Consultants

Notes:

TOM = Task order manager
RPM = Remedial project manager
QA = Quality assurance
GCAL = Gulf Coast Analytical

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SAP WORKSHEETS #34 — 36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE

(UFP-QAPP Manual Section 5.2.1), (UFP-QAPP Manual Section 5.2.2), (Figure 37 UFP-QAPP Manual), (Table 9 UFP-QAPP Manual)

Data Review Input	Description	Responsible for Verification (name, organization)	Internal/ External
Verification (Step I) Chain-of-custody forms Sample Login/ Receipt	Review the sample shipment for completeness, integrity, and sign accepting the shipment. All sample labels will be checked against the chain-of-custody form, and any discrepancies will be identified, investigated, and corrected. The samples will be logged in at every storage area and work station required by the designated analyses. Individual analysts will verify the completeness and accuracy of the data recorded on the forms.	Laboratory sample custodians and analysts, GCAL Laboratory	Internal
Verification (Step I) Chain-of-custody forms	Check that the chain-of-custody form was signed/dated by the sampler relinquishing the samples and by the laboratory sample custodian receiving the samples for analyses.	Project chemist or data validators, Resolution Consultants	External
Verification (Step I) SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	FOL or designee, Resolution Consultants	Internal
Verification (Step I) Sample log sheets and field notes	Verify that information recorded in the log sheets and field notes are accurate and complete.	FOL or designee, Resolution Consultants	Internal
Verification (Step I) Field QC samples	Check that field QC samples, described in Worksheet #12 and listed in Worksheet #20 were collected as required.	FOL or designee, Resolution Consultants	Internal
Verification (Step I) Analytical data package	Verify all analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory project manager (or designee) will sign the case narrative for each data package.	Laboratory project manager, GCAL Laboratory	Internal
Verification (Step I) Analytical data package	Verify the data package for completeness. Missing information will be requested from the laboratory and validation (if performed) will be suspended until missing data are received.	FOL, Project chemist or data validators, Resolution Consultants	External
Verification (Step I) Electronic data deliverables	Verify the electronic data against the chain-of-custody and hard copy data package for accuracy and completeness.	Data manager and/or validator, Resolution Consultants	External
Validation (Step IIa) Chain-of-custody	Examine the traceability of the data from time of sample collection until reporting of data. Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Project chemist or data validators, Resolution Consultants	External
Validation (Step IIa) Holding Times	Review that the samples were shipped and stored 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. If holding times were not met, confirm that deviations were documented.	Project chemist or data validators, Resolution Consultants	External

Data Review Input	Description	Responsible for Verification (name, organization)	Internal/ External
Validation (Steps IIa/IIb) Laboratory data results for accuracy	Ensure that the laboratory QC samples were analyzed and that the measurement performance criteria, listed in Worksheet #28, were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed, as listed in Worksheet #12, and that the analytical QC criteria were met.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Field and laboratory duplicate analyses for 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/LCSDs. Ensure compliance with the precision goals listed in Worksheet #12 and 28.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Sample results for 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.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Project action limits	Assess and document the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as undetected.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Data quality assessment report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Present tabular qualified data and data qualifier codes and summarize data qualification outliers. Determine if the data met the measurement performance criteria and determine the impact of any deviations on the technical usability of the data.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) SAP QC sample documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within acceptance limits.	Project chemist or data validators, Resolution Consultants	External
Validation (Step IIb) Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and laboratory SOP requirements and matrix interferences effect on the analytical results.	Project chemist or data validators, Resolution Consultants	External
Validation (Step IIb) Project quantitation limits for sensitivity	Ensure that the project detection limits were achieved.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Soil and Groundwater — VOCs	Assess VOC data using SW846 8260B method-specific criteria, the DoD QSM, and those listed in Worksheets # 12, 19, and 28. *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, (October 1999) will be used to apply qualifiers to data.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Soil and Groundwater — SVOCs and PAHs via SIM	Assess SVOC and PAHs via SIM data using SW846 8270D method-specific criteria, the DoD QSM, and those listed in Worksheets # 12, 19, and 28. *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, (October 1999) will be used to apply qualifiers to data.	Project chemist or data validators, Resolution Consultants	External

Data Review Input	Description	Responsible for Verification (name, organization)	Internal/ External
Validation (Steps IIa/IIb) Soil and Groundwater — Pesticides	Assess pesticide data using SW846 8081A method-specific criteria, the DoD QSM, and those listed in Worksheets # 12, 19, and 28. *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, (October 1999) will be used to apply qualifiers to data.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Soil and Groundwater — PCBs	Assess PCB data using SW846 8082A method-specific criteria, the DoD QSM, and those listed in Worksheets # 12, 19, and 28. *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, (October 1999) will be used to apply qualifiers to data.	Project chemist or data validators, Resolution Consultants	External
Validation (Steps IIa/IIb) Soil and Groundwater — Metals	Assess Metals data using U.S. EPA Methods 6020A, 7470a, and 7471B method-specific criteria, the DoD QSM, and those listed in Worksheets # 12, 19, and 28. *U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, (October 2004) will be used to apply qualifiers to data.	Project chemist or data validators, Resolution Consultants	External

Notes:

SAP = Sampling and analysis plan
FOL = Field operations leader
QC = Quality control
RPD = Relative percent difference
MS/MSD = Matrix spike/Matrix Spike duplicate
LCS/LCSD = Laboratory control sample/laboratory control sample duplicate
SOP = Standard operating procedure
VOC = Volatile organic compounds
DoD QSM = *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010
U.S. EPA = U.S Environmental Protection Agency
SVOC = Semi-volatile organic compounds
PAH = Polynuclear aromatic hydrocarbons
SIM = Selective ion monitoring
PCBs = Polychlorinated biphenyl compounds

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SAP WORKSHEET #37: USABILITY ASSESSMENT

(UFP-QAPP Manual Section 5.2.3)

Data Review

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

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

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

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

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

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

- Sensitivity — The Project Chemist, acting on behalf of the Project Team, will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described.

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

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

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

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

Identify the personnel responsible for performing the usability assessment:
The Resolution Consultants TOM, Project Chemist, and FOL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.

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Appendix A
Applicable Technical Procedures

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

GUIDANCE

Title: **Design and Installation of Monitoring Wells**

Effective Date: January 29, 2013	**Number:** SESDGUID-101-R1

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDGUID-101-R1, *Design and Installation of Monitoring Wells*, replaces SESDPROC-101-R0. **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Danny France. The FQM was changed from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Section 1.4:** Replaced the "SESD Operating Procedure for Field Records and Documentation, SESDPROC-204-Most Recent Version" with its updated version, the "SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version. **Section 1.5.1:** Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used. **Section 1.5.2:** On the second bullet, replaced the reference with the "SESD Operating Procedure for Logbooks (SESDPROC-010)."	January 29, 2013
SESDGUID-101-R0, *Design and Installation of Monitoring Wells*, Original Issue	February 18, 2008

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when designing and installing permanent and temporary groundwater monitoring wells to be used for collection of groundwater samples.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when designing, constructing and installing groundwater monitoring wells. On the occasion that SESD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used for any aspect of the design, construction and/or installation of a groundwater monitoring well, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Science and Ecosystem Support Division, Region 4, Athens, GA, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Groundwater Sampling, SESDPROC-301, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

EPA/540/S-95/503, *Nonaqueous Phase Liquids Compatibility with Materials Used in Well Construction, Sampling, and Remediation*

ASTM standard D5092, *Design and Installation of Ground Water Monitoring Wells in Aquifers*

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when constructing and installing groundwater monitoring wells. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy (SHEMP) Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate. Section 2.6, Safety Procedures for Drilling Activities, contains detailed and specific safety guidelines that must be followed by Branch personnel when conducting activities related to monitoring well construction and installation.

1.5.2 Procedural Precautions

The following precautions should be considered when constructing and installing groundwater monitoring wells.

- Special care must be taken to minimize or prevent inadvertent cross-contamination between borehole locations. Equipment, tools and well materials must be cleaned and/or decontaminated according to procedures found in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).
- All field activities are documented in a bound logbook according to the procedures found in SESD Operating Procedure for Logbooks (SESDPROC-010).

2 Permanent Monitoring Well Design Considerations

2.1 General

The design and installation of permanent monitoring wells involves drilling into various types of geologic formations that exhibit varying subsurface conditions. Designing and installing permanent monitoring wells in these geologic environments may require several different drilling methods and installation procedures. The selection of drilling methods and installation procedures should be based on field data collected during a hydrogeologic site investigation and/or a search of existing data. Each permanent monitoring well should be designed and installed to function properly throughout the duration of the monitoring program. When designing monitoring wells, the following should be considered:

- Short-and long-term objectives;
- Purpose of the well(s);
- Probable duration of the monitoring program;
- Contaminants likely to be monitored;
- Surface and subsurface geologic conditions;
- Properties of the aquifer(s) to be monitored;
- Well screen placement;
- General site conditions; and
- Potential site health and safety hazards.

In designing permanent monitoring wells, the most reliable, obtainable data should be utilized. Once the data have been assembled and the well design(s) completed, a drilling method(s) must be selected. The preferred drilling methods for installing monitoring wells are those that temporarily case the borehole during drilling and the construction of the well, e.g. hollow-stem augers and sonic methods. However, site conditions or project criteria may not allow using these methods. When this occurs, alternate methods should be selected that will achieve the project objectives. The following discussion of methods and procedures for designing and installing monitoring wells will cover the different aspects of selecting materials and methods, drilling boreholes, and installing monitoring devices.

2.2 Drilling Methods

The following drilling methods may be used to install environmental monitoring wells or collect samples under various subsurface conditions. In all cases the preferred methods are those that case the hole during drilling, i.e. Hollow Stem Augers (HSA) and sonic methods using an override system. Other methods may be used where specific subsurface or project criteria dictate.

2.2.1 Hollow Stem Auger (HSA)

This type of auger consists of a hollow, steel stem or shaft with a continuous, spiraled steel flight, welded onto the exterior. A hollow auger bit, generally with carbide teeth, disturbs soil material when rotated, whereupon the spiral flights transport the cuttings to the surface. This method is best suited in soils that have a tendency to collapse when disturbed. A monitoring well can be installed inside of hollow-stem augers with little or no concern for the caving potential of the soils. If caving sands exist during monitoring well installations, a drilling rig must be used that has enough power to extract the augers from the borehole without having to rotate them. A bottom plug, trap door, or pilot bit assembly can be used at the bottom of the augers to keep out most of the soils and/or water that have a tendency to enter the bottom of the augers during drilling. Potable water (analyzed for contaminants of concern) may be poured into the augers during drilling to equalize pressure so that the inflow of formation materials will be held to a minimum. Water-tight center bits are not acceptable because they create suction when extracted from the augers. This suction forces or pulls cuttings and formation materials into the augers, defeating the purpose of the center plug. Augering without a center plug or pilot bit assembly is permitted, provided that the soil plug, formed in the bottom of the augers, is removed before sampling or installing well casings. Removing the soil plug from the augers can be accomplished by drilling and washing out the plug using a rotary bit, or augering out the plug with a solid-stem auger bit sized to fit inside the hollow-stem auger. Bottom plugs can be used where no soil sampling is conducted during the drilling process. The bottom plug is wedged into the bottom of the auger bit and is knocked out at depth with drill pipe or the weight of the casing and screen assembly. The plug material should be compatible with the screen and casing materials. The use of wood bottom plugs is not acceptable. The type of bottom plug, trap door, or pilot bit assembly proposed for the drilling activity should be approved by a senior field geologist prior to drilling operations. Boreholes can be augered to depths of 150 feet or more (depending on the auger size), but generally boreholes are augered to depths less than 100 feet.

2.2.2 Solid Stem Auger

This type of auger consists of a sealed hollow or solid stem or shaft with a continuous spiraled steel flight welded on the outside of the stem. An auger bit connected to the bottom disturbs soil material when rotated and the helical flights transport cuttings to the surface. At the desired depth the entire auger string is removed to gain access to the bottom of the borehole. This auger method is used in cohesive and semi-cohesive soils that do not have a tendency to collapse when disturbed. Boreholes can be augered to depths of 200 feet or more (depending on the auger size), but generally boreholes are augered to depths less than 100 feet.

Both of the previously discussed auger methods can be used in unconsolidated soils and semi-consolidated (weathered rock) soils, but not in competent rock.

Each method can be employed without introducing foreign materials into the borehole such as water and drilling fluids, minimizing the potential for cross contamination. Minimizing the risk of cross contamination is one of the most important factors to consider when selecting the appropriate drilling method(s) for a project.

2.2.3 Sonic Methods

These methods generally alternately advance concentric hollow drill stems using rotation in conjunction with axial vibration of the drill stem. After each stage of drill stem advancement, the inner string is removed with a core of drill cuttings while the outer 'override' string remains to hold the borehole open. The cuttings can be removed nearly intact from the inner casing for examination of the stratigraphy prior to sampling or disposal. Because there are no auger flights to increase the borehole diameter, the quantity of cuttings removed from the hole is minimized as compared to hollow stem augering. With moderate rotation, smearing of the formation materials on the borehole walls is reduced as well. This drilling method is useful in a variety of materials, from flowing sands to heavily consolidated or indurated formations.

In flowing sands, the drill casings can be filled and/or pressurized with potable water to prevent excess entry of formation materials into the drill string. The same QA/QC requirements for sampling of material introduced to the borehole apply as in other drilling methods. Because the amount of water introduced into the borehole can be significant, an approximation of the water used in the drilling process should be logged for use in estimating appropriate well development withdrawal.

Sonic drilling allows a larger diameter temporary casing to be set into a confining layer while drilling proceeds into deeper aquifers. This temporary casing is then removed during the grouting operation. In many cases this will be acceptable technique. However, the level of contamination in the upper aquifer, the importance of the lower aquifers for drinking water uses, the permeability and continuity of the confining layer, and state regulations should be taken into account when specifying this practice as opposed to permanent outer casing placed into the confining unit. Note that when using the temporary casing practice, it is critical that grout be mixed and placed properly as specified elsewhere in this section.

Because the total borehole diameter in sonic drilling is only incrementally larger than the inner casing diameter, particular care should be taken that the well casing is placed in the center of the drill stem while placing the filter pack. Centralizers should be used in most cases to facilitate centering, particularly in the case of deep wells with PVC casing.

2.2.4 Rotary Methods

These methods consist of a drill pipe or drill stem coupled to a drilling bit that rotates and cuts through the soils. The cuttings produced from the rotation of the drilling bit are transported to the surface by drilling fluids which generally consist of water, drilling mud, or air. The water, drilling mud, or air are forced down through the drill pipe, and out through the bottom of the drilling bit. The cuttings are then lifted to the surface between the borehole wall and the drill pipe, (or within a concentric drill stem in reverse rotary). Except in the case of air rotary, the drilling fluid provides a hydrostatic pressure that reduces or prevents borehole collapse. When considering this method, it is important to evaluate the potential for contamination when fluids and/or air are introduced into the borehole.

Due to the introduction of the various circulating fluids, the use of rotary methods requires that the potential for contamination by these fluids be evaluated. Water and mud rotary methods present the possibility of trace contamination of halogenated compounds when municipal water supplies are used as a potable water source. Air rotary drilling can introduce contamination through the use of lubricants or entrained material in the air stream. Unless contaminated formations are cased off, the circulation of drilling fluids presents a danger of cross contamination between formations. In any of the rotary (or sonic) methods, care must be exercised in the selection and use of compounds to prevent galling of drill stem threads.

2.2.4.1 Water Rotary

When using water rotary, potable water (that has been analyzed for contaminants of concern) should be used. If potable water (or a higher-quality water) is not available on-site, then potable water will have to be transported to the site or an alternative drilling method will have to be selected. Water does not clog the formation materials, but the suspended drilling fines can be carried into the formation, resulting in a very difficult to develop well. This method is most appropriate for setting isolation casing.

2.2.4.2 Air Rotary

Air rotary drilling uses air as a drilling fluid to entrain cuttings and carry them to the surface. High air velocities, and consequently large air volumes and compressor horsepower are required. “Down-the-hole” (DTH) percussion hammers driven by the air stream can be used with this method to rapidly penetrate bedrock materials. Where a casing through unconsolidated material is required to prevent borehole collapse, it can be driven in conjunction with advancement of the drill stem.

When using air rotary drilling in any zone of potential contamination, the cuttings exiting the borehole must be controlled. This can be done using

the dual-tube reverse circulation method where cuttings are carried to the surface inside dual-wall drill pipe and separated with a cyclone separator. An air diverter with hose or pipe carrying cuttings to a waste container is also an acceptable alternative. Allowing cuttings to blow uncontrolled from the borehole is not acceptable.

When using air rotary, the issue of contaminants being introduced into the borehole by the air stream must be addressed. Screw compressor systems should have a coalescing filter system in good working order to capture excess entrained compressor oils. The lubricant to be used with DTH hammers as well as thread lubricants to be used on drill stem should be evaluated for their potential impact on analytical samples.

2.2.4.3 Mud Rotary

Mud rotary is an undesirable drilling method because contamination can be introduced into the borehole from the constituents in the drilling mud, cross contamination can occur along the borehole column, and it is difficult to remove the drilling mud from the borehole after drilling and during well development. The drilling mud can also carry contaminates from a contaminated zone to an uncontaminated zone thereby cross-contaminating the borehole. If mud rotary is selected, only potable water and pure (no additives) bentonite drilling muds should be used. All materials used should have adequate documentation as to manufacturer's recommendations and product constituents. QA/QC samples of drilling muds and potable water should be sampled at a point of discharge from the circulation system to assure that pumps and piping systems are not contributing cross-contamination from previous use.

2.2.5 Other Methods

Other methods such as the cable-tool method, jetting method, and boring (bucket auger) method are available. If these and/or other methods are selected for monitoring well installations, they should be approved by a senior field geologist before field work is initiated.

2.3 Borehole Construction

2.3.1 Annular Space

The borehole or hollow stem auger should be of sufficient diameter so that well construction can proceed without major difficulties. For open boreholes, the annular space should be approximately 2" to allow the uniform deposition of well materials around the screen and riser, and to allow the passage of tremie pipes and well materials without unduly disturbing the borehole wall. For example, a 2" nominal diameter (nom.) casing would require a 6" inside diameter (ID) borehole.

In hollow stem augers and sonic method drill casing, the ID should be of sufficient size to allow the passage of the tremie pipe to be used for well grout placement, as well as free passage of filter sands or bentonite pellets dropped through the auger or casing. In general, 4-1/4" ID should be the minimum size used for placement of 2" nom. casing and 8-1/4" ID for 4" nom. casing. Larger augers should be used where installation difficulties due to geologic conditions or greater depths are anticipated, e.g. larger augers might be required to place a bentonite pellet seal through a long water column.

2.3.2 Over-drilling the Borehole

Sometimes it is necessary to over-drill the borehole in anticipation of material entering the augers during center bit removal or knocking out of the bottom plug. Normally, 3 to 5 feet is sufficient for over-drilling. The borehole can also be over-drilled to allow for an extra space or a "sump" area below the well screen. This "sump" area provides a space to attach a 5 or 10 foot section of well casing to the bottom of the well screen. The extra space or "sump" below the well screen serves as a catch basin or storage area for sediment that flows into the well and drops out of suspension. These "sumps" are added to the well screens when the wells are screened in aquifers that are naturally turbid and will not yield clear formation water (free of visible sediment) even after extensive development. The sediment can then be periodically pumped out of the "sump" preventing the well screen from clogging or "silting up". If the borehole is inadvertently drilled deeper than desired, it can be backfilled to the design depth with bentonite pellets, chips, or the filter sand that is to be used for the filter pack.

2.3.3 Filter Pack Placement

When placing the filter pack into the borehole, a minimum of 6-inches of the filter pack material should be placed under the bottom of the well screen to provide a firm base. Also, the filter pack should extend a minimum of 2-feet above the top of the well screen to allow for settling and to isolate the screened interval from the grouting material. In open boreholes, the filter pack should be placed by the tremie or positive displacement method. Placing the filter pack by pouring the sand into an open drill stem is acceptable with the use hollow stem augers, and other methods where the borehole is temporarily cased down to the filter pack.

2.3.4 Filter Pack Seal – Bentonite Pellet Seal (Plug)

Bentonite pellets consist of ground, dried bentonite compacted into pellets available in several sizes. Bentonite pellets are compressed to a bulk density of 70-80 lbs/ft^3 and hydrate to a 30% min. solids material. Where neat cement grouts are to be used, the placement of a bentonite pellet seal above the filter pack is mandatory to prevent the possibility of grout infiltration into the screened interval prior to setting. Bentonite chips or other sealing products should not be

substituted in this application. Where bentonite grouts are to be used, the placement of a bentonite pellet seal is optional, but desirable.

Since bentonite pellets begin hydrating rapidly, they can be very difficult to place properly. They are generally placed by pouring slowly into open boreholes, hollow stem augers or sonic drill pipe. In some cases, pellets are placed by tremie pipe and flushed into place with potable water. A tamper can be used to ensure that the material is being placed properly and to rapidly break up any pellet bridging that occurs.

Pellet seals should be designed for a two-foot thickness of dry pellets above the filter pack. Hydration may extend the height of the seal. Where neat cement grouts are to be used, the pellets should be hydrated for eight hours, or the manufacturer's recommended hydration time, whichever is greater. Where the water table is temporarily below the pellet seal, potable (or higher quality) water should be added repeatedly to hydrate the pellets prior to grouting.

2.3.5 Grouting the Annular Space

The annular space between the casing and the borehole wall should be filled with either a 30% solids bentonite grout, a neat cement grout, or a cement/bentonite grout. Each type of grout selected should be evaluated as to its intended use and integrity. Bentonite grouts are preferred unless the application dictates the use of another material.

Bentonite grout shall be a 30% solids pure bentonite grout. Drilling muds are not acceptable for grouting. The grout should be placed into the borehole, by the tremie method, from the top of the bentonite seal to within 2-feet of the ground surface or below the frost line, whichever is the greater depth. The bentonite pellet seal or filter pack should not be disturbed during grout placement, either by the use of a side discharge port on the tremie tube, or by maintaining clearance between the bottom of the tremie tube and the bentonite seal or filter pack. The grout should be allowed to cure for a minimum of 24 hours before the concrete surface pad is installed. The preferred method of achieving proper solids content is by measurement of ingredients per the manufacturer's specifications during mixing with a final check by grout balance after mixing. Bentonite grouts should have a minimum density of 10 lbs/gal to ensure proper gelling and low permeability. The density of the first batch of grout should be measured while mixing to verify proper measurement of ingredients. In addition, the grouting operation should not cease until the bentonite grout flowing out of the borehole has a minimum density of 10 lbs/gal. Estimating the grout density is not acceptable.

Cement grouts are generally dictated where a high level of dissolved solids or a particular dissolved constituent would prevent proper gelling of a bentonite grout. Neat cement grouts (cement without additives) should be mixed using 6 gallons of

water per 94-lb bag of Type 1 Portland cement to a density of 15lbs/gal. The addition of bentonite (5 to 10 percent) to the cement grout can be used to delay the "setting" time and may not be needed in all applications. The specific mixtures and other types of cement and/or grout proposed should be evaluated on a case by case basis by a senior field geologist.

2.3.6 Above Ground Riser Pipe and Outer Casing

The well casing, when installed and grouted, should extend above the ground surface a minimum of 2.5 feet. A vent hole should be drilled into the top of the well casing cap to permit pressure equalization, if applicable. An outer protective casing should be installed into the borehole after the annular grout has cured for at least 24 hours. The outer protective casing should be of steel construction with a hinged, locking cap. Generally, outer protective casings used over 2-inch well casings are 4 inches square by 5 feet long. Similarly, protective casings used over 4-inch well casings are 6 inches square and 5 feet long. Other types of protective casing including those constructed of pipe are also acceptable. All protective casings should have sufficient clearance around the inner well casings, so that the outer protective casings will not come into contact with the inner well casings after installation. The protective casings should have a weep hole to allow drainage of accumulated rain or spilled purge water. The weep hole should be approximately 1/4-inch in diameter and drilled into the protective casings just above the top of the concrete surface pad to prevent water from standing inside of the protective casings. Protective casings made of aluminum or other soft metals are normally not acceptable because they are not strong enough to resist tampering. Aluminum protective casing may be used in very corrosive environments such as coastal areas.

Prior to installing the protective casing, the bentonite grout in the borehole annulus is excavated to a depth of approximately two feet. The protective casing is installed by pouring concrete into the borehole on top of the grout. The protective casing is then pushed into the wet concrete and borehole a minimum of 2 feet. Extra concrete may be needed to fill the inside of the protective casing so that the level of the concrete inside of the protective casing is at or above the level of the surface pad. In areas where frost heave of the surface pad is possible, the protective casing should first be pressed into the top surface of the bentonite grout seal and concrete poured around the protective casing. A granular material such as sand or gravel can then be used to fill the space between the riser and protective casing. The use of granular material instead of concrete between the protective casing and riser will also facilitate the future conversion of the well to a flush-mount finish, if required. The protective casing should extend above the ground surface to a height so that the top of the inner well casing is exposed when the protective casing is opened. At each site, all locks on the outer protective casings should be keyed alike.

2.3.7 Concrete Surface Pad

A concrete surface pad should be installed around each well at the same time as the outer protective casing is being installed. The surface pad should be formed around the well casing. Concrete should be placed into the pad forms and into the borehole (on top of the grout) in one operation making a contiguous unit. The size of the concrete surface pad is dependent on the well casing size. If the well casing is 2 inches in diameter, the pad should be 3 feet x 3 feet x 4 inches. If the well casing is 4 inches in diameter, the pad should be 4 feet x 4 feet x 6 inches. Round concrete surface pads are also acceptable. The finished pad should be slightly sloped so that drainage will flow away from the protective casing and off of the pad. A minimum of one inch of the finished pad should be below grade to prevent washing and undermining by soil erosion.

2.3.8 Surface Protection – Bumper Guards

If the monitoring wells are located in a high traffic area, a minimum of three bumper guards consisting of steel pipes 3 to 4 inches in diameter and a minimum 5-foot length should be installed. These bumper guards should be installed to a minimum depth of 2 feet below the ground surface in a concrete footing and extend a minimum of 3 feet above ground surface. Concrete should also be placed into the steel pipe to provide additional strength. Substantial steel rails and/or other steel materials can be used in place of steel pipe. Welding bars between the bumper posts can provide additional strength and protection in high traffic areas, but the protective bumpers should not be connected to the protective casing.

2.4 Construction Techniques

2.4.1 Well Installation

The borehole should be bored, drilled, or augered as close to vertical as possible, and checked with a plumb bob or level. Deviation from plumb should be within 1° per 50ft of depth. Slanted boreholes are undesirable and should be noted in the boring logs and final construction logs. The depth and volume of the borehole, including the over-drilling if applicable, should have been calculated and the appropriate materials procured prior to drilling activities.

The well casings should be secured to the well screen by flush-jointed threads and placed into the borehole and plumbed by the use of centralizers and/or a plumb bob and level. Another method of placing the well screen and casings into the borehole and plumbing them at the same time is to suspend the string of well screen and casings in the borehole by means of a hoist on the drill rig. This wireline method is especially useful if the borehole is deep and a long string of well screen and casings have to be set and plumbed.

No lubricating oils or grease should be used on casing threads. No glue of any type should be used to secure casing joints. Teflon "O" rings can also be used to insure a tight fit and minimize leakage; however, "O" rings made of other materials are not acceptable if the well is going to be sampled for organic compound analyses.

Before the well screen and casings are placed on the bottom of the borehole, at least 6 inches of filter material should be placed at the bottom of the borehole to serve as a firm footing. The string of well screen and casings should then be placed into the borehole and plumbed. Centralizers can be used to plumb a well, but centralizers should be placed so that the placement of the filter pack, bentonite pellet seal, and annular grout will not be hindered. Centralizers placed in the wrong locations can cause bridging during material placement. Monitoring wells less than 50 feet deep generally do not need centralizers. If centralizers are used they should be placed below the well screen and above the bentonite pellet seal. The specific placement intervals should be decided based on site conditions.

When installing the well screen and casings through hollow-stem augers, the augers should be slowly extracted as the filter pack, bentonite pellet seal, and grout are tremied and/or poured into place. The gradual extraction of the augers will allow the materials being placed in the augers to flow out of the bottom of the augers into the borehole. If the augers are not gradually extracted, the materials (sand, pellets, etc.) will accumulate at the bottom of the augers causing potential bridging problems.

After the string of well screen and casing is plumb, the filter pack material should then be placed around the well screen to the designated depth. With cased drilling methods, the sand should be poured into the casing or augers until the lower portion is filled. The casing or augers are then withdrawn, allowing the sand to flow into the evacuated space. With hollow stem augers, sand should always fill the augers 6-12 inches, maintained by pouring the sand while checking the level with a weighted tag line. The filter pack sand in open boreholes should be installed by tremie methods, using water to wash the sand through the pipe to the point of placement.

After the filter pack has been installed, the bentonite pellet seal (if used) should be placed directly on top of the filter pack to an unhydrated thickness of two feet. When installing the seal for use with cement grouts, the bentonite pellet seal should be allowed to hydrate a minimum of eight hours or the manufacturer's recommended hydration time, whichever is longer.

After the pellet seal has hydrated for the specified time, the grout should then be pumped by the tremie method into the annular space around the casings. The grout should be allowed to set for a minimum of 24 hours before the surface pad and protective casing are installed.

After the surface pad and protective casing are installed, bumper guards should be installed (if needed). The bumper guards should be placed around the concrete surface pad in a configuration that provides maximum protection to the well. Each piece of steel pipe or approved material should be installed into an 8-to 10-inch diameter hole, to a minimum depth of 2 feet below ground surface, and filled with concrete. As previously stated, the bumper guard should extend above the ground surface a minimum of 3 feet. The total length of each bumper guard should be a minimum of 5 feet.

After the wells have been installed, the outer protective casing should be painted with a highly visible paint. The wells should be permanently marked with the well number, date installed, site name, elevation, etc., either on the cover or an appropriate place that will not be easily damaged and/or vandalized.

If the monitoring wells are installed in a high traffic area such as a parking lot, in a residential yard, or along the side of a road it may be desirable to finish the wells to the ground surface and install water-tight flush mounted traffic and/or man-hole covers. Flush mounted traffic and man-hole covers are designed to extend from the ground surface down into the concrete plug around the well casing. Although flush mounted covers may vary in design, they should have seals that make the unit water-tight when closed and secured. The flush mounted covers should be installed slightly above grade to minimize standing water and promote runoff. Permanent identification markings should be placed on the covers or in the concrete plug around the cover. Expansive sealing plugs should be used to cap the well riser to prevent infiltration of any water that might enter the flush cover.

2.4.2 Double-Cased Wells

Double-cased wells should be constructed when there is reason to believe that interconnection of two aquifers by well construction may cause cross-contamination or when flowing sands make it impossible to install a monitoring well using conventional methods. A highly contaminated surface soil zone may also be cased off so that drilling may continue below the casing with reduced danger of cross contamination. A pilot borehole should be bored through the overburden and/or the contaminated zone into the clay confining layer or bedrock. An outer casing (sometimes called surface or pilot casings) should then be placed into the borehole and sealed with grout. The borehole and outer casing should extend into tight clay a minimum of two feet and into competent bedrock a minimum of 1 foot. The total depths into the clay or bedrock will vary, depending on the plasticity of the clay and the extent of weathering and\or fracturing of the bedrock. The final depths should be approved by a senior field geologist. The size of the outer casing should be of sufficient inside diameter to contain the inner casing, and the 2-inch minimum annular space. In addition, the borehole should be of sufficient size to contain the outer casing and the 2-inch minimum outer annular space, if applicable.

The outer casing should be grouted by the tremie, displacement, grout shoe, or Halliburton method from the bottom to the ground surface. The grout should be pumped into the annular space between the outer casing and the borehole wall. A minimum of 24 hours should be allowed for the grout plug (seal) to cure before attempting to drill through it. The grout mixture used to seal the outer annular space should be either a neat cement, cement/bentonite, cement/sand, or a 30% solids bentonite grout. However, the seal or plug at the bottom of the borehole and outer casing should consist of a Type I portland cement/bentonite or cement/sand mixture. The use of a pure bentonite grout for a bottom plug or seal is not acceptable, because the bentonite grout cures to a gel-like material, and is not rigid enough to withstand the stresses of drilling. When drilling through the seal, care should be taken to avoid cracking, shattering, or washing out the seal. If caving conditions exist so that the outer casing cannot be sufficiently sealed by grouting, the outer casing should be driven into place and a grout seal placed in the bottom of the casing.

2.4.2.1 Bedrock Wells

The installation of monitoring wells into bedrock can be accomplished in two ways:

1. The first method is to drill or bore a pilot borehole through the soil overburden into the bedrock. An outer casing is then installed into the borehole by setting it into the bedrock, and grouting it into place as described in the previous section. After the grout has set, the borehole can then be advanced through the grout seal into the bedrock. The preferred method of advancing the borehole into the bedrock is rock coring. Rock coring makes a smooth, round hole through the seal and into the bedrock without cracking and/or shattering the seal. Roller cone bits are used in soft bedrock, but extreme caution should be taken when using a roller cone bit to advance through the grout seal in the bottom of the borehole because excessive water and "down" pressure can cause cracking, eroding (washing), and/or shattering of the seal. Low volume air hammers may be used to advance the borehole, but they have a tendency to shatter the seal because of the hammering action. If the structural integrity of the grout seal is in question, a pressure test can be utilized to check for leaks. A visual test can also be made by examining the cement/concrete core that is collected when the seal is cored with a diamond coring bit. If the seal leaks (detected by pressure testing) and/ or the core is cracked or shattered, or if no core is recovered because of washing, excessive down pressure, etc., the seal is not acceptable. The concern over the structural integrity of the grout seal applies to all double cased wells. Any proposed method of double casing and/or seal testing will be evaluated on its own merits, and will have to be approved by a senior field geologist before and during drilling activities, if

applicable. When the drilling is complete, the finished well will consist of an open borehole from the ground surface to the bottom of the well. There is no inner casing, and the outer surface casing, installed down into bedrock, extends above the ground surface, and also serves as the outer protective casing. If the protective casing becomes cracked or is sheared off at the ground surface, the well is open to direct contamination from the ground surface and will have to be repaired immediately or abandoned. Another limitation to the open rock well is that the entire bedrock interval serves as the monitoring zone. In this situation, it is very difficult or even impossible to monitor a specific zone, because the contaminants being monitored could be diluted to the extent of being non-detectable. The installation of open bedrock wells is generally not acceptable in the Superfund and RCRA programs, because of the uncontrolled monitoring intervals. However, some site conditions might exist, especially in cavernous limestone areas (karst topography) or in areas of highly fractured bedrock, where the installation of the filter pack and its structural integrity are questionable. Under these conditions the design of an open bedrock well may be warranted.

2. The second method of installing a monitoring well into bedrock is to install the outer surface casing and drill the borehole (by an approved method) into bedrock, and then install an inner casing and well screen with the filter pack, bentonite seal, and annular grout. The well is completed with a surface protective casing and concrete pad. This well installation method gives the flexibility of isolating the monitoring zone(s) and minimizing inter-aquifer flow. In addition, it gives structural integrity to the well, especially in unstable areas (steeply dipping shales, etc.) where the bedrock has a tendency to shift or move when disturbed. Omitting the filter pack around the well screen is a general practice in some open rock borehole installations, especially in drinking water and irrigation wells. However, without the filter pack to protect the screened interval, sediment particles from the well installation and/or from the monitoring zone could clog the well screen and/or fill the screened portion of the well rendering it inoperable. Also, the filter pack serves as a barrier between the bentonite seal and the screened interval. Rubber inflatable packers have been used to place the bentonite seal when the filter pack is omitted, but the packers have to remain in the well permanently and, over a period of time, will decompose and possibly contribute contaminants to the monitoring zone.

2.5 Well Construction Materials

2.5.1 Introduction

Well construction materials are chosen based on the goals and objectives of the proposed monitoring program and the geologic conditions at the site(s). In this section, the different types of available materials will be discussed.

2.5.2 Well Screen and Casing Materials

When selecting the materials for well construction, the prime concern should be to select materials that will not contribute foreign constituents, or remove contaminants of concern from the ground water. If the monitoring program is designed to analyze for organic compounds, stainless steel materials are the preferred choice. If the monitoring program calls for the analyses of only inorganic compounds or the contaminants or formation are highly corrosive, then rigid PVC materials meeting National Sanitary Foundation (NSF) Standard 14 type WC (Well Casing) are acceptable. PVC materials may be acceptable for monitoring identified organic compounds in a soluble aqueous phase where incompatibilities are known to not exist. EPA document EPA/540/S-95/503, *Nonaqueous Phase Liquids Compatibility with Materials Used in Well Construction, Sampling, and Remediation* (http://www.epa.gov/ada/-download/issue/napl.pdf) should be used for guidance in this area and in the use of PVC with non-aqueous phase liquids (NAPLs). Another concern is to select materials that will be rugged enough to endure the entire monitoring period. Site conditions will generally dictate the type of materials that can be used. A preliminary field investigation should be conducted to determine the geologic conditions, so that the most suitable materials can be selected. The best grade or highest quality material for that particular application should be selected. Each manufacturer can supply the qualitative data for each grade of material that is being considered. All materials selected for monitoring well installation should be evaluated and approved by a senior field geologist prior to field activities.

Well screen and casing materials generally used in monitoring well construction on RCRA and Superfund sites are listed in order of preference:

1. Stainless Steel (304 or 316)
2. Rigid PVC meeting NSF Standard 14 (type WC)
3. Other (where applicable)

There are other materials used for well screens and casings such as black iron, carbon steel, galvanized steel, and fiberglass, but these materials are not recommended for use in long term monitoring programs at hazardous waste sites, because of their low resistance to chemical attack and potential constituent contribution to the ground water. In cases where a driven casing is used, or a high strength outer casing is needed, carbon steel may be acceptable in non-corrosive aquifers. This outer casing should have threaded connections. Welding casing is

not an acceptable practice unless all relevant safety issues have been adequately addressed.

The minimum nominal casing size for most permanent monitoring wells will be 2". Where a complete program of installation, monitoring, and abandonment is being designed, smaller wells may be installed if suitable purging and sampling equipment for the smaller diameter wells can be specified and obtained. The length of well screens in permanent monitoring wells should be long enough to effectively monitor the interval or zone of interest. However, well screens designed for long term monitoring purposes should normally not be less than 5 feet in length. Well screens less than 5 feet long are generally only used in temporary monitoring wells where ground water samples are collected for screening purposes.

2.5.3 Filter Pack Materials

The filter pack materials should consist of clean, rounded to well-rounded, hard, insoluble particles of siliceous composition. The required grain-size distribution or particle sizes of the filter pack materials should be selected based upon a sieve analysis conducted on the soil samples collected from the aquifer materials and/or the formation(s) to be monitored. Filter pack materials should not be accepted unless proper documentation can be furnished as to the composition, grain-size distribution, cleaning procedure, and chemical analysis. If a data search reveals that there is enough existing data to adequately design the well screen and filter pack, then it may not be necessary to conduct a sieve analysis on the formation materials to be monitored. However, all data and design proposals will be evaluated and approved by a senior staff geologist before field activities begin.

2.5.4 Filter Pack and Well Screen Design

The majority of monitoring wells are installed in shallow ground water aquifers that consist of silts, clays, and sands in various combinations. These shallow aquifers are not generally characteristic of aquifers used for drinking water. Therefore, modifications to the procedures used for the design of water well filter packs may be required. In cases where insufficient experience exists with local or similar materials, the filter pack and well screen design should be based on the results of a sieve analysis conducted on soil samples collected from the aquifer or the formation(s) that will be monitored.

In formations consisting primarily of fines (silts and clays), the procedures for water well screen design may result in requirements for filter packs and screen slot sizes that are not available. In those cases the selection of 0.010" screen slots with a 20-40 sand filter pack, or 0.005" screen slots with 100 sand filter pack for very fine formations, will be acceptable practice. Table 6.6.1 provides size specifications for the selection of sand packs for fine formation materials. ASTM standard D5092, *Design and Installation of Ground Water Monitoring Wells in*

Aquifers, may be consulted for further guidance on specifications for sand appropriate for these applications.

Table 6.6.1
Sand Pack Specifications

Screen Opening (in)	Sand Pack Mesh Name	1% Passing Size (d-1) (in)	10% Passing Size (d-10) (in)	30% Passing Size (d-30) (in)	Derived 60% Passing Size (d-60) (in)	Range for Uniformity Coefficient
0.005-0.006	100	.0035 - .0047	.0055 - .0067	.0067 - .0083	.0085 - .0134	1.3 - 2.0
0.010"	20-40	.0098 - .0138	.0157 - .0197	.0197 -.0236	.020 - .0315	1.1 - 1.6

The following procedure should be used in coarser grained formations:

The data from the sieve analysis are plotted on a grain-size distribution graph, and a grain-size distribution curve is generated. From this grain-size distribution curve, the uniformity coefficient (Cu) of the aquifer material is determined. The Cu is the ratio of the 60 percent finer material (d60) to the 10 percent finer material (d10)

$$Cu = (d60/d10)$$

The Cu ratio is a way of grading or rating the uniformity of grain size. For example, a Cu of unity means that the individual grain sizes of the material are nearly all the same, while a Cu with a large number indicates a large range of particle sizes. As a general rule, a Cu of 2.5 or less should be used in designing the filter pack and well screen.

Before designing the filter pack and well screen, the following factors should be considered:

1. Select the well screen slot openings that will retain 90 percent of the filter pack material.

2. The filter pack material should be of the size that minimizes head losses through the pack and also prevents excessive sediment (sand, silt, clay) movement into the well.

3. A filter material of varying grain sizes is not acceptable because the smaller particles fill the spaces between the larger particles thereby reducing the void spaces and increasing resistance to flow. Therefore, filter material of the same grain size and well rounded is preferred.

4. The filter pack design is based on the gradation of the finest aquifer materials being analyzed.

Steps to design a filter pack in aquifers:

1. Construct a grain-size distribution curve, on a grain-size distribution graph, from the sieve analysis of the aquifer materials. The filter pack design (as stated above) is based on the gradation of the finest aquifer materials.

2. Multiply the d30 size from the grain-size distribution graph by a factor of four to nine (Pack-Aquifer ratio). A factor of four is used if the formation is fine-grained and uniform (Cu is less than 3), six if it is coarse-grained and non-uniform, and up to nine if it is highly non-uniform and contains silt. Head losses through filter packs increase as the Pack-Aquifer (P-A) ratios decrease. In order to design a fairly stable filter pack with a minimum head loss, the d30 size should be multiplied by a factor of four.

3. Plot the point from step 2 on the d30 abscissa of a grain-size distribution graph and draw a smooth curve with a uniformity coefficient of approximately 2.5.

4. A curve for the permissible limits of the filter pack is drawn plus or minus 8 per cent of the desired curve with the Cu of 2.5.

5. Select the slot openings for the well screen that will retain 90 per cent or more of the filter pack material.

The specific steps and procedures for sieve analysis and filter pack design can be found in soil mechanics, ground water, and water well design books. The staff geologists and/or engineers should be responsible for the correct design of the monitoring wells and should be able to perform the design procedures.

2.6 Safety Procedures for Drilling Activities

A site health and safety plan should be developed and approved by the Branch Safety Officer or designee prior to any drilling activities, and should be followed during all drilling activities. The driller or designated safety person should be responsible for the safety of the drilling team performing the drilling activities. All personnel conducting drilling activities should be qualified in proper drilling and safety procedures. Before any drilling activity is initiated, utilities should be marked or cleared by the appropriate state or municipal utility protection organization. In developed areas, additional measures

should be taken to locate utilities not covered by the utility protection program. Before operating the drill rig, a pilot hole should be dug (with hand equipment) to a depth of three feet to check for undetected utilities or buried objects. Proceed with caution until a safe depth is reached where utilities normally would not be buried. The following safety requirements should be adhered to while performing drilling activities:

1. All drilling personnel should wear safety hats, safety glasses, and steel toed boots. Ear plugs are required and will be provided by the safety officer or driller.

2. Work gloves (cotton, leather, etc.) should be worn when working around or while handling drilling equipment.

3. All personnel directly involved with the drilling rig(s) should know where the kill switch(s) is located in case of emergencies.

4. All personnel should stay clear of the drill rods or augers while in motion, and should not grab or attempt to attach a tool to the drill rods or augers until they have completely stopped rotating. Rod wipers, rather than gloves or bare hands should be used to remove mud, or other material, from drill stem as it is withdrawn from the borehole.

5. Do not hold drill rods or any part of the safety hammer assembly while taking standard penetration tests or while the hammer is being operated.

6. Do not lean against the drill rig or place hands on or near moving parts at the rear of the rig while it is operating.

7. Keep the drilling area clear of any excess debris, tools, or drilling equipment.

8. The driller will direct all drilling activities. No work on the rig or work on the drill site will be conducted outside of the driller's direction. Overall drill site activities will be in consultation with the site geologist or engineer, if present.

9. Each drill rig will have a first-aid kit and a fire extinguisher located on the rig in a location quickly accessible for emergencies. All drilling personnel will be familiarized with their location.

10. Work clothes will be firm fitting, but comfortable and free of straps, loose ends, strings etc., that might catch on some moving part of the drill rig.

11. Rings, watches, or other jewelry will not be worn while working around the drill rig.

12. The drill rig should not be operated within a minimum distance of 20 feet of overhead electrical power lines and/or buried utilities that might cause a safety hazard. In addition, the drill rig should not be operated while there is lightening in the area of the drilling site. If an electrical storm moves in during drilling activities, the area will be vacated until it is safe to return.

2.7 Well Development

A newly completed monitoring well should not be developed for at least 24 hours after the surface pad and outer protective casing are installed. This will allow sufficient time for the well materials to cure before development procedures are initiated. The main purpose of developing new monitoring wells is to remove the residual materials remaining in the wells after installation has been completed, and to try to re-establish the natural hydraulic flow conditions of the formations which may have been disturbed by well construction, around the immediate vicinity of each well. A new monitoring well should be developed until the column of water in the well is free of visible sediment, and the pH, temperature, turbidity, and specific conductivity have stabilized. In most cases the above requirements can be satisfied; however, in some cases the pH, temperature, and specific conductivity may stabilize but the water remains turbid. In this case the well may still contain well construction materials, such as drilling mud in the form of a mud cake and/or formation soils that have not been washed out of the borehole. Excessive or thick drilling mud cannot be flushed out of a borehole with one or two well volumes of flushing. Continuous flushing over a period of several days may be necessary to complete the well development. If the well is pumped to dryness or near dryness, the water table should be allowed to sufficiently recover (to the static water level) before the next development period is initiated. Caution should be taken when using high rate pumps and/or large volume air compressors during well development because excessive high rate pumping and high air pressures can damage or destroy the well screen and filter pack. The onsite geologist should make the decision as to the development completion of each well. All field decisions should be documented in the field log book.

The following development procedures, listed in approximate increasing order of the energy applied to the formation materials, are generally used to develop wells:

1. Bailing
2. Pumping/overpumping
3. Surging
4. Backwashing ("rawhiding")
5. Jetting
6. Compressed air (with appropriate filtering): airlift pumping and air surging

These development procedures can be used, individually or in combination, in order to achieve the most effective well development. In most cases, over-pumping and surging will adequately develop the well without imparting undue forces on the formation or well materials. Except when compressed air is being used for well development, sampling can be initiated as soon as the ground water has re-equilibrated, is free of visible sediment, and the water quality parameters have stabilized. Since site conditions vary, even between wells, a general rule-of-thumb is to wait 24 hours after development to sample a new monitoring well. Wells developed with stressful measures may require as long as a 7-day interval before sampling. In particular, air surge developed wells require 48 hours or longer after development so that the formation can dispel the compressed air and re-stabilize to pre-well construction conditions. Because of the danger of introducing

contaminants with the airstream, the possibility of entraining air in the aquifer, and the violent forces imparted to the formation, air surging is the least desired method of development and should only be used where there is a specific need for the procedure. Air-lift pumping is permissible where an eductor pipe is used and several well volumes of water are removed from the well by other by pumping means after air-lift pumping. The selected development method(s) should be approved by a senior field geologist before any well installation activities are initiated.

2.8 Well Decommisioning (Abandonment)

When a decision is made to decommission (abandon) a monitoring well, the borehole should be sealed in such a manner that the well cannot act as a conduit for migration of contaminants from the ground surface to the water table or between aquifers. To properly decommission a well, the preferred method is to completely remove the well casing and screen from the borehole, clean out the borehole, and backfill with a cement or bentonite grout, neat cement, or concrete. In order to comply with state well decommissioning requirements, the appropriate state agency should be notified (if applicable) of monitoring well decommissioning. However, some state requirements are not explicit, so a technically sound well abandonment method should be designed based on the site geology, well casing materials, and general condition of the well(s).

2.8.1 Decommissioning Procedures

As previously stated the preferred method should be to completely remove the well casing and screen from the borehole. This may be accomplished by augering with a hollow-stem auger over the well casing down to the bottom of the borehole, thereby removing the grout and filter pack materials from the hole. The well casing should then be removed from the hole with the drill rig. The clean borehole can then be backfilled with the appropriate grout material. The backfill material should be placed into the borehole from the bottom to the top by pressure grouting with the positive displacement method (tremie method). This abandonment method can be accomplished on small diameter (1-inch to 4-inch) wells without too much difficulty. With wells having 6-inch or larger diameters, the use of hollow-stem augers for casing removal is very difficult or almost impossible. Instead of trying to ream the borehole with a hollow-stem auger, it is more practical to force a drill stem with a tapered wedge assembly or a solid-stem auger into the well casing and extract it out of the borehole. Wells with little or no grouted annular space and/or sound well casings can be removed in this manner. However, old wells with badly corroded casings and/or thickly grouted annular space have a tendency to twist and/or break-off in the borehole. When this occurs, the well will have to be grouted with the remaining casing left in the borehole. The preferred method in this case should be to pressure grout the borehole by placing the tremie tube to the bottom of the well casing, which will be the well screen or the bottom sump area below the well screen. The pressurized grout will be forced out through the well screen into the filter material and up the inside of the well casing sealing holes and breaks that are present.

A PVC well casing may be more difficult to remove from the borehole than a metal casing, because of its brittleness. If the PVC well casing breaks during removal, the borehole should be cleaned out by using a drag bit or roller cone bit with the wet rotary method to grind the casing into small cuttings that will be flushed out of the borehole by water or drilling mud. Another method is to use a solid-stem auger with a carbide tooth pilot bit to grind the PVC casing into small cuttings that will be brought to the surface on the rotating flights. After the casing materials have been removed from the borehole, the borehole should be cleaned out and pressure grouted with the approved grouting materials.

Where state regulations and conditions permit, it may be permissible to grout the casing in place. This decision should be based on confidence in the original well construction practice, protection of drinking water aquifers, and anticipated future property uses. The pad should be demolished and the area around the casing excavated. The casing should be sawn off at a depth of three feet below ground surface. The screen and riser should be tremie grouted with a 30% solids bentonite grout in the saturated zone. The remaining riser may be grouted with a cement grout for long term resistance to desiccation.

3 Temporary Monitoring Well Installation

3.1 Introduction

Five types of temporary monitoring well installation techniques have been demonstrated as acceptable. The type selected for a particular site is dependent upon site conditions. The project leader and site geologist should be prepared to test temporary well installations on site and select the best solution. Temporary wells are cost effective, may be installed quickly, and provide a synoptic picture of ground water quality.

Temporary monitoring well locations are not permanently marked, nor are their elevations normally determined. Sand pack materials may or may not be used, but typically there is no bentonite seal, grout, surface completion, or extensive development (as it normally applies to permanent monitoring wells). Temporary wells are generally installed, purged, sampled, removed, and backfilled in a matter of hours.

Due to the nature of construction, turbidity levels may initially be high. However, these levels may be reduced by low flow purging and sampling techniques as described in Section 7.2.4.

Temporary wells may be left overnight, for sampling the following day, but the well must be secured, both against tampering and against the fall hazard of the open annulus. If the well is not sampled immediately after construction, the well should be purged prior to sampling as specified in SESD Operating Procedure for Groundwater Sampling, SESDPROC-301.

3.2 Data Limitation

Temporary wells described in this section are best used for delineation of contaminant plumes at a point in time, and for some site screening purposes. They are not intended to replace permanent monitoring wells. Temporary wells can be used in conjunction with a mobile laboratory, where quick analytical results can be used to delineate contaminant plumes.

3.3 Temporary Well Materials

Materials used in construction of temporary monitoring wells are the same standard materials used in the construction of permanent monitoring wells. Sand used for the filter pack (if any) should be as specified in Section 2.5.3, Filter Pack Materials. The well screen and casing should be stainless steel for ruggedness and suitability for steam cleaning and solvent rinsing. Other materials may be acceptable, on a case by case basis. Some commercially available temporary well materials, pre-packed riser, screen and filter pack assemblies are available commercially; however, these pre-assembled materials cannot be cleaned. Appropriate QA/QC must be performed to assure there will be no introduction of contamination.

3.4 Temporary Monitoring Well Borehole Construction

Borehole construction for temporary wells is as specified in Section 2.3, using a drill rig. Alternatively, boreholes may be constructed using hand augers or portable powered augers (generally limited to depths of ten feet or less). If a drill rig is used to advance the borehole, the augers must be pulled back the length of the well screen (or removed completely) prior to sampling. When hand augers are used, the borehole is advanced to the desired depth (or to the point where borehole collapse occurs). In situations where borehole collapse occurs, the auger bucket is typically left in the hole at the point of collapse while the temporary well is assembled. When the well is completely assembled, a final auger bucket of material is quickly removed and the well is immediately inserted into the borehole, pushing, as needed, to achieve maximum penetration into the saturated materials.

3.5 Temporary Monitoring Well Types

Five types of monitoring wells which have been shown to be acceptable are presented in the order of increasing difficulty to install and increasing cost:

3.5.1 No Filter Pack

This is the most common temporary well and is very effective in many situations. After the borehole is completed, the casing and screen are simply inserted. This is the least expensive and fastest well to install. This type of well is extremely sensitive to turbidity fluctuations because there is no filter pack. Care should be taken to not disturb the casing during purging and sampling.

3.5.2 Inner Filter Pack

This type differs from the "No Filter Pack" well in that a filter pack is placed inside the screen to a level approximately 6 inches above the well screen. This ensures that all water within the casing has passed through the filter pack. For this type well to function properly, the static water level must be at least 6-12 inches above the filter pack. The screen slots may plug in some clayey environments with this construction method and others that use sand only inside the well screen.

3.5.3 Traditional Filter Pack

For this type of well, the screen and casing are inserted into the borehole, and the sand is poured into the annular space surrounding the screen and casing. Occasionally, it may be difficult to effectively place a filter pack around shallow open boreholes, due to collapse. This method requires more sand than the "inner filter pack" well, increasing material costs. As the filter pack is placed, it mixes with the muddy water in the borehole, which may increase the amount of time needed to purge the well to an acceptable level of turbidity.

3.5.4 Double Filter Pack

The borehole is advanced to the desired depth. As with the "inner filter pack" the well screen is filled with filter pack material and the well screen and casing inserted until the top of the filter pack is at least 6 inches below the water table. Filter pack material is poured into the annular space around the well screen. This type temporary well construction can be effective in aquifers where fine silts or clays predominate. This construction technique takes longer to implement and uses more filter pack material than others previously discussed.

3.5.5 Well-in-a-Well

The borehole is advanced to the desired depth. At this point, a 1-inch well screen and sufficient riser is inserted into a 2-inch well screen with sufficient riser, and centered. Filter pack material is then placed into the annular space surrounding the 1-inch well screen, to approximately 6 inches above the screen. The well is then inserted into the borehole.

This system requires twice as much well screen and riser, with attendant increases in assembly and installation time. The increased amount of well construction materials results in a corresponding increase in decontamination time and costs. The use of pre-packed well screens in this application will require rinse blanks of each batch of screens. Pre-pack Screen assemblies cannot be decontaminated for reuse.

3.6 Decommissioning

Temporary well boreholes must be decommissioned after sampling and removal of the screen and riser. Backfilling the holes with cuttings may be acceptable practice for shallow holes in uniform materials with expected low contamination levels. Use of cuttings would not be an acceptable practice if waste materials were encountered or a confining layer was breached. Likewise, where the borehole is adjacent to, or downgradient of contaminated areas, the loose backfilled material could create a highly permeable conduit for contaminant migration. If the borehole will not be backfilled with the soil cuttings for this or other reasons, then SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, should be referenced regarding disposal of the cuttings as IDW.

4 Temporary Monitoring Well Installation Using the Geoprobe® Screen Point 15/16 Groundwater Sampler

4.1 Introduction

The Geoprobe® Screen Point 15/16 Groundwater Sampler is a discrete interval ground water sampling device that can be pushed to pre-selected sampling depths in saturated, unconsolidated materials. Once the target depth has been reached, the screen is opened and groundwater can be sampled as a temporary monitoring well, which yields a representative, uncompromised sample from that depth. Using knock-out plugs, this method also allows for grouting of the push hole during sample tool retrieval.

The Screen Point® 15 sampler consist of four parts (drive point, screen, sampler sheath and drive head), with an assembled length of 52 inches (1321 mm) and a maximum OD of 1.5 inches (38 mm). When opened, it has an exposed screen length of 41 inches (1041 mm). It is typically pushed using 1.25-inch probe rod. The Screen Point® 16 consists of the same parts and works in the same fashion, the only differences being larger diameter and its use with 1.5" rods.

4.1.1 Assembly of Screen Point® 15/16 Groundwater Sampler

1. Install O-ring on expendable point and firmly seat in the angled end of the sampler sheath.

2. Place a grout plug in the lower end of the screen section. Grout plug material should be chosen with consideration for site specific Data Quality Objectives (DQOs).

3. When using stainless steel screen, place another O-ring* in the groove on the upper end of the screen and slide it into the sampler sheath.

4. Place an O-ring* on the bottom of the drive head and thread into the top of the sampler sheath.

5. The Screen Point® 15/16 Groundwater Sampler is now assembled and ready to push for sample collection.

   * It should be noted that O-ring use in steps 3 and 4 are optional.

4.1.2 Installation of Screen Point® 15/16 Groundwater Sampler

1. Attach drive cap to top of sampler and slowly drive it into the ground. Raise the hammer assembly, remove the drive cap and place an O-ring* in the top groove of the drive head. Add a probe rod and continue to push the rod string.

2. Continue to add probe rods until the desired sampling depth is reached.

3. When the desired sampling depth is reached, re-position the probe derrick and position either the casing puller assembly or the rod grip puller over the top of the top probe rod.

4. Thread a screen push adapter on an extension rod and attach sufficient additional extension rods to reach the top of the Screen Point® 15/16 sampler. Add an extension handle to the top of the string of extension rods and run this into the probe rod, resting the screen push adapter on top of the sampler.

5. To expose the screened portion of the sampler, exert downward pressure on the sampler, using the extension rod and push adapter, while pulling the probe rod upward. To expose the entire open portion of the screen, pull the probe rod upward approximately 41 inches.

6. At this point, the Screen Point® 15/16 Groundwater Sampler has been installed as a temporary well and may be sampled using appropriate ground water sampling methodology. If waters levels are less than approximately 25 feet, EIB personnel typically use a peristaltic pump, utilizing low-flow methods, to collect ground water samples from these installations. If water levels are greater than 25 feet, a manual bladder pump, a micro bailer, or other method may have to be utilized to collect the sample (SESD Operating Procedure for Groundwater Sampling, SESDPROC-301-R0) provides detailed descriptions of these techniques and methods).

4.1.3 Special Considerations for Screen Point® 15/16 Installations

<u>Decommissioning (Abandonment)</u>

In many applications, it may be appropriate to grout the abandoned probe hole where a Screen Point® 15/16 sampler was installed. This probe hole decommissioning can be accomplished through two methods which are determined by location and contamination risk. In certain non-critical areas, boreholes may be decommissioned by filling the saturated zone with bentonite pellets and grouting the vadose zone with neat cement poured from the surface or Bentonite pellets properly hydrated in place. Probe holes in areas where poor borehole sealing could present a risk of contaminant migration should be decommissioned by pressure grouting through the probe rod during sampler retrieval. To accomplish this, the grout plug is knocked out of the bottom of the screen using a grout plug push adapter and a grout nozzle is fed through the probe rod, extending just below the bottom of the screen. As the probe rod and sampler

are pulled, grout is injected in the open hole below the screen at a rate that just fills the open hole created by the pull. Alternatively, the screen can generally be pulled and the hole re-probed with a tool string to be used for through-the-rod grouting.

Screen Material Selection

Screen selection is also a consideration in sampling with the Screen Point® 15/16 sampler. The screens are available in two materials, stainless steel and PVC. Because of stainless steel's durability, ability to be cleaned and re-used, and overall inertness and compatibility with most contaminants, it is the material typically used during EIB investigations.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Equipment Inventory and Management**

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator (DCC).

History	Effective Date
SESDPROC-108-R4, *Equipment Inventory and Management,* replaces SESDPROC-108-R3. **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the EIB Branch Chief from Archie Lee to Danny France. Changed the EAB Branch Chief From Bill Cosgrove to John Deatrick. Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement - Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted references to the H: drive. **Section 1.4.7**: Defined SESD Field Equipment Tracking System. **Section 2.4.1**: Modified statement to read - Maintenance, calibration and verification requirements for equipment will be documented in the SESD Field Equipment Tracking System or equipment log. **Section 2.4.2**: Modified the statement to read - The BFEMs are responsible for placing all documentation associated with the equipment repair in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. Modified the statement to read - The BFEM or qualified individual is responsible for placing all documentation associated with the repair of equipment stored at the SESD laboratory in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. **Section 2.5.1**: All calibrations or calibration verifications performed at the SESD laboratory or Field Equipment Center will be recorded in the SESD Field Equipment Tracking System or appropriate equipment log. **Section 2.5.2:** Modified statement to read - Thermometers used during field investigations will be verified by the SESD Analytical Support Branch (ASB) or EPA contract personnel utilizing the ASB Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G. **Section 2.7:** Modified statement to read - This will be done using the SESD Field Equipment Tracking System or by using an equipment sign-out/sign-in form.	February 6, 2013

SESDPROC-108-R3, *Equipment Inventory and Management*, replaces SESDPROC-108-R2. **General Information** Previous versions of the document will be maintained by the Document Control Coordinator (DCC). Changed from Field Quality Manager (FQM) to DCC. **Title Page** Changed EIB Branch Chief from Antonio Quinones to Archie Lee. **Section 1.3** Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD H: drive and for maintaining records of review conducted prior to its issuance. **Section 1.6** Added reference to Analytical Support Branch Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G. **Section 2.2.2** Removed requirement that the BFEM will maintain copy of all paperwork associated with the purchase of equipment. **Section 2.3.1** Removed requirement that the BFEM send the current copy of the equipment inventory to the FQM. The BFEM will maintain the current list of equipment subject to the procedure. Modified #9 to state that the inventory list will include a reference to the manufacturer's instruction manual title. **Section 2.3.2** Removed requirement that FQM will ensure that duplicate identification numbers are not assigned to equipment. BFEMs will be responsible for ID numbers. **Section 2.4.2** Removed requirement that the BFEM must notify the FEC personnel via email that the repaired equipment has been returned. **Section 2.6, #2** Acceptance record will be a copy of the signed invoice.	April 6, 2009
SESDPROC-108-R2, *Equipment Inventory and Management*, replaces SESDPROC-108-R1. **General** Changed title on cover page for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. **Section 1.6** Corrected form number for Load-In Form from SESDFORM-012 to SESDFORM-	October 19, 2007

011. **Section 2.4.2** Corrected form number for Load-In Form from SESDFORM-012 to SESDFORM-011. Added last paragraph regarding adversely impacted data. **Section 2.5** Added second sentence regarding pos-calibration verification checks for equipment. Added forth and fifth sentences regarding calibration acceptance criteria. Added second paragraph pertaining to outside calibration services. **Section 2.3.1** Added item 9.	
SESDPROC-108-R1, *Equipment Inventory and Management*, replaces SESDPROC-108-R0. **Section 1.1** Modified purpose to include equipment that may come into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations **Section 1.2** Modified scope to include microscopes, volumetric equipment and equipment that may come into direct contact with the sample media (i.e. submersible pumps) and has the potential to cross contaminate samples between sampling stations **Section 1.3** Modified requirements for maintaining official copy of procedure. **Section 1.5** Combined section 1.5.1 and 1.5.2. Added "maintenance" to list of manufacturer's directions to follow. Added that a copy of the instruction manual will be provided with sampling equipment to be taken in the field. **Section 2.1** Added equipment that comes in direct contact with sample media to list of equipment covered under the procedure. Added reference to decontamination procedures. **Section 2.2** Renamed Section 2.2 to include equipment receipt. Added Section 2.2.2 (Receiving Equipment). Section 2.2.1 - Modified and expanded the purchase of equipment procedure. Removed reference to SESDPROC-015. **Section 2.3** Incorporated inventory procedures from SESDPROC-104 to this section. Added Section 2.3.1 -Equipment Labeling and Marking. **Section 2.4** Added critical spare parts will be sent out with equipment. Section 2.4.2 - Changed email notification to written notification. Included reference to SESDFORM-011. Added procedures for equipment that require software upgrades.	October 1, 2007

Section 2.5 Included visual inspection of equipment. Added reference to equipment that is not directly used for field measurement and added that calibration and measurement requirements are found in individual measurement procedures. Added maintenance procedure for microscopes and volumetric equipment. Changed title of Section 2.5.2 to include certification and verification records. Changed storage location of NIST Certificates. Defined NIST as National Institute of Standards and Technology. Added language to verify the quality of equipment. Added procedure for the verification of thermometers. Section 2.5.3 – Added that records of calibration will be maintained in equipment log books and procedure for returning equipment unsuitable for use. **Section 2.6** Changed requirement from the original Purchase Order to a copy of the order will be maintained by the branch field equipment managers. **Section 2.8** Removed Section from operating procedure **Section 2.9** Removed Section 2.9 from operating procedure. Information on the glove and bottle return policy was added to Section 2.6 of SESDPROC-011-R1.	
SESDPROC-108-R0, Equipment and Supply Management, Original Issue.	May 4, 2007

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Contents

1 General Information

1.1 Purpose

This document describes the procedures necessary to demonstrate the operational status and inventory of equipment used for field measurement activities and equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations.

1.2 Scope/Application

This procedure covers the approaches and documentation used for the purchase, maintenance, calibration, verification and inventory of equipment used for direct field measurement activities. This procedure includes the maintenance and use of microscopes, volumetric equipment and equipment that may come into direct contact with the sample media (i.e. submersible pumps) and has the potential to cross contaminate samples between sampling stations. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Calibration

The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system and corresponding known values. The results of a calibration permit the estimation of errors associated with the measurement equipment.

1.4.2 Calibration Verification

Provides a means of determining that deviations between measured values and known values are within the limits of error defined during calibration. The results provide an indication that the instrument/system is working properly.

1.4.3 Preventative Maintenance

A program of routine actions such as cleaning, lubrication, adjusting, or testing to keep equipment ready for use. The most important effect of a preventative maintenance program is to ensure measurement system reliability.

1.4.4 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager (BFEM) will be designated for the Enforcement and Investigations Branch (EIB) and the Ecological Assessment Branch (EAB).

1.4.5 Qualified Individual

Individual who has received on the job training and has experience working with specific measurement instruments.

1.4.6 Equipment Log

Notebook, log book or electronic file that contains a copy of the purchase order, if available, as well as, maintenance, calibration, verification records, performance checks, correction factors and sign-out/sign-in records. Equipment logs will be established for all equipment used for field measurement activities or equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations. The development of equipment logs is the responsibility of the Branch Field Equipment Managers.

1.4.7 SESD Field Equipment Tracking System

Electronic database used to sign-out/sign-in sampling equipment, track equipment use history, and maintain equipment availability status.

1.5 Precautions

To ensure the safe and reliable operation of equipment, the manufacturers' directions for transport, cleaning, decontamination, storage, maintenance and operation of equipment

will be followed. When possible, a copy of the instruction manual will be provided with sampling equipment to be taken in the field. In addition, field activities will be conducted in accordance to the SESD Safety, Health and Environmental Management Program (SHEMP).

Handling of contaminated equipment is addressed under the following operating procedures: Field Equipment Cleaning and Decontamination at the Field Equipment Center (SESDPROC-206) and Field Equipment Cleaning and Decontamination (SESDPROC-205).

1.6 References

American National Standard ANSI/ASQC M1-1996, *Calibration Systems*

American National Standard ANSI/NCLS Z2540-1-1994, *Calibration Laboratories, and Measuring and Test Equipment - General Requirements*

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, most recent version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, most recent version.

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, most recent version.

SESD Operating Procedure for Competency and Proficiency Testing, SESDPROC-006, most recent version.

SESD Operating Procedure for Purchasing of Services and Supplies, SESDPROC-015, most recent version.

SESD Operating Procedure for Control of Nonconforming Work, SESDPROC-019, most recent version.

FEC Loan-In Form, SESDFORM-011, most recent version.

Analytical Support Branch Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G.

2 Methodology

2.1 General

Equipment (hardware and related software) used for field measurement activities and equipment that comes into direct contact with the sample media and has the potential to cross contaminate samples between sampling stations will meet quality requirements identified for each piece of equipment. Important factors in establishing quality requirements include the parameters to be measured and the sensitivity and specificity of the detection system used. Quality requirements must include ensuring that equipment is ready for use. Specifically:

1. SESD will have adequate equipment to conduct measurement activities.

2. Equipment used for field measurement activities will be capable of achieving the accuracy and precision required by the measurement objectives.

3. When SESD is required to use environmental data collection equipment outside its permanent control, it will ensure that all relevant SESD criteria in this procedure are met.

4. Field measurement equipment used by SESD will be secured at all times, as feasible, based on field conditions.

5. Operating instructions and/or manuals from the manufacturer will be available for each piece of equipment, when possible.

6. Field investigators will only operate equipment for procedures which they are authorized per SESDPROC-006 to perform.

7. Equipment used for field measurements will be handled, transported, shipped, stored and operated in a manner that prevents damage, gross contamination and deterioration. Equipment will be handled and maintained in accordance to the manufacturer's operating instructions. Decontamination of equipment will be in accordance to procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) and Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206).

2.2 Equipment Purchase and Receipt

2.2.1 Purchasing Equipment

Equipment covered in this procedure is purchased using procurement requests (PR) or purchase card orders and can be initiated by the BFEM or designee. The initiator of a purchase is responsible for preparing a PR or purchase card order that specifies the correct name of the item, the relevant quality criteria of the item, the item number, a quote or price, possible sources and any other associated information that helps identify the correct item to be purchased. The sources identified must be capable of providing the equipment according to the defined specifications. A copy of the purchase order or purchase card order will be maintained in the equipment log. Once the order has been prepared, the information is forwarded to the appropriate BFEM for origination of the purchase and management will authorize and sign the order and forward it to the purchasing official. The purchasing official will ensure the order is processed according to the information on the PR or purchase card order.

2.2.2 Receiving Equipment

Anyone can accept delivery of equipment orders. For equipment purchases received at the SESD laboratory, the BFEM or initiator of the purchase will inspect the items to ensure they comply with the PR or purchase card order. If the items are acceptable, the BFEM or initiator will sign and date the invoice that accompanied the order and any other required documentation and forward it to the purchasing official. The purchasing official will maintain a file of all SESD purchases.

For purchases received at the Field Equipment Center, contract personnel will inspect the items to ensure they comply with the PR or purchase card order. If the items are acceptable, the contract personnel will sign and date the invoice that accompanied the order and any other required documentation and forward it to the appropriate BFEM. The original paperwork (signed invoice) will be forwarded to the purchasing official.

If any equipment is found to be unsuitable for use, the BFEMs will document the problems and any action taken to correct that problem. The documentation will include a description of the item, the deficiency and the vendor. The BFEM will also compile all occurrences of unsuitable consumables, supplies or services and determine what further corrective action may be necessary and notify the Field Quality Manager if deemed necessary.

2.3 Equipment Inventory

2.3.1 Inventory

The BFEMs are responsible for maintaining a current equipment inventory list for their specific areas. The BFEMs will maintain a central, comprehensive list of all field measurement and sampling equipment subject to this procedure. Field measurement equipment, including sampling equipment that comes into contact with the sample media and has the potential to cross contaminate samples between stations (i.e., submersible pumps and vacuum chambers), will be included in this inventory. The equipment inventory list for each instrument or piece of equipment will include:

1. A description of the property and software, if applicable (e.g., pH meter, dissolved oxygen meter, etc.).
2. Manufacturer or vendor name
3. Model number
4. Serial number or other manufacturer identification number
5. A unique identifier, known as the SESD ID Number. These identifiers will be assigned by the BFEM.
6. Storage location (e.g., Field Equipment Center, SESD Laboratory)
7. Date received and/or date placed in service, where available.
8. Status of the equipment (e.g., surplus, damaged, etc.)
9. A reference to the manufacturer's instruction manual title.

2.3.2 Equipment Labeling and Marking

Prior to being placed into the equipment inventory, all equipment covered under this procedure will be inventoried and labeled with a unique SESD identification number. The identification number will be assigned only to the identified equipment and will not be reused if the meter is excised or disposed. Equipment that is no longer used or cannot be repaired will be removed from the inventory.

Equipment received after the effective date of this procedure will be assigned a number that includes the date the equipment was received followed by a sequential number starting at 01 (e.g., 031407-01, 031407-02, etc.). BFEMS will ensure that duplicate SESD ID Numbers are not assigned to equipment.

Equipment received before the effective date of this operating procedure will be assigned a number that will incorporate all or part of the serial number or other manufacturer number as the unique SESD ID number.

2.4 Equipment Maintenance

Maintenance will consist of preventative care and corrective repair. Both approaches should be used to keep equipment in working order. Each item of field measurement equipment will be checked by a qualified individual prior to use. Critical spare parts, which cannot be easily obtained while in the field, will be sent out with the equipment during field investigations.

Equipment maintenance will include software upgrades for certain instruments. SESD management will assign staff to serve as a point of contact for equipment, such as GPS or Sondes that may require periodic software upgrades in order to collect field measurements. The point of contact will ensure that software upgrades are conducted on all equipment. The point of contacts will maintain a list of the equipment which will include the serial number, SESD ID Number, software version, software upgrade dates and the storage location of the equipment. The point of contact will provide the information to the appropriate BFEM to be included in the equipment inventory.

2.4.1 Documentation

The BFEMs are responsible for the development of equipment logs. Maintenance, calibration and verification requirements for equipment will be documented in the SESD Field Equipment Tracking System or equipment log.

2.4.2 Repair and Re-certification for Use

Equipment known or suspected to be defective will be taken out of service and clearly labeled, preferably with a red tag, until it has been repaired and shown by calibration, verification or testing to function properly. When equipment is tagged in the field as defective, the SESD project leader will notify the appropriate BFEM in writing, either by email or by using a load-in form (SESDFORM-011), that equipment requires repair.

For equipment maintained at the FEC which is routinely handled and maintained by contract personnel, the BFEM will be responsible for having the equipment repaired. Once repaired, the BFEM will notify the FEC contract personnel that the equipment has been returned. The contract personnel will ensure that the equipment is functioning properly prior to it being used for field investigations. The BFEMs are responsible for placing all documentation associated with the equipment repair in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. This documentation includes written notifications regarding the equipment repair and any information provided from the manufacturer which describes the defect and the repair.

For equipment stored at the SESD laboratory, or stored at the FEC and not routinely handled and maintained by contract personnel, the BFEM or a designated qualified individual will be responsible for having the equipment repaired. Once repaired, the BFEM or qualified individual will ensure the equipment is functioning properly prior to it being used for field investigations. The BFEM or qualified individual is responsible for placing all documentation associated with the repair of equipment stored at the SESD laboratory in the SESD Field Equipment Tracking System or appropriate maintenance section of the equipment log. This documentation includes the written notifications regarding the equipment repair and any information provided which describes the defect and the repair.

If there is the potential that data collected with defective equipment were adversely impacted, the BFEM will notify the appropriate Branch Quality Assurance Officer (QAO). The QAO will notify affected management and The Field Quality Manager and the issue will be handled according to the SESD Operating Procedure for Control of Nonconforming Work (SESDPROC-019).

2.4.3 Required Maintenance

The BFEMs are responsible for ensuring that equipment maintenance is performed in-house or by an outside service.

2.4.4 Record Keeping

Records of all maintenance, service, repairs and histories of any damage, malfunction or modification of field measurement equipment will be maintained in the equipment logs. The record will describe hardware and software changes and/or updates and show the dates when these occurred.

2.5 Equipment Calibration

Prior to being used in the field, measurement equipment will be visually inspected and a calibration, calibration verification or performance check will be conducted to ensure it is in working condition. A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. When practicable, equipment requiring calibration will be labeled to indicate the status of calibration and the date when re-calibration is due. Calibration acceptance criteria based on data quality objectives will be established on a case by case basis for each field study. Project Leaders will be responsible for ensuring the acceptance criteria are met. Calibration and measurement requirements are found in individual measurement procedures.

When equipment or reference standards are calibrated by an outside calibration service, SESD will ensure the vendor is ISO 17025 accredited or has some other equivalent means for demonstrating competence, measurement capability and traceability.

All reasonable measures will be taken to safeguard equipment from adjustments which would invalidate the measurements following calibration of the equipment. Field investigators will handle, transport and store equipment in accordance to manufacturer recommendations. Periodic calibration or performance checks will be conducted during field investigations to ensure that equipment calibration has not been adversely impacted between measurement locations.

For equipment that is not directly used for field measurement, visual examination, safety checks or, if appropriate, performance checks, will be conducted by the FEC contract personnel or the project leader to ensure the working condition of the equipment. Microscopes, including attachments, will be cleaned and serviced as needed prior to use.

Volumetric equipment will be maintained by visual examination and cleaning at the Field Equipment Center. Performance checks will be conducted by the FEC contract personnel or the project leader prior to initial use and at intervals depending on the frequency of use.

2.5.1 Documentation

Records for equipment that is sent off-site for calibration recertification will be maintained in the equipment log books. All calibrations or calibration verifications performed at the SESD laboratory or Field Equipment Center will be recorded in the SESD Field Equipment Tracking System or appropriate equipment log.

All calibrations or calibration verifications performed in the field will be recorded in the project specific logbook(s) by the individual conducting the calibration or verification and stored in the associated project file. Calibration of equipment conducted by SESD field investigators and other qualified personnel will be conducted in accordance with the manufacturer's recommendations

When calibrations require correction factors, the correction factors will be documented in instrument operating procedures, equipment logs and written methods (field logbooks).

2.5.2 Traceability, Certification, and Verification Records

The program for the calibration or calibration verification of equipment must ensure that, where the concept is applicable, all significant measurements are traceable through certificates of calibration held by SESD, and/or to National

Standards of Measurement. National Institute of Standards and Technology (NIST) traceable standards will be used, if available. For equipment maintained at the FEC and sent off-site for calibration, certificates or other records of calibration will be maintained in a central file at the FEC. Additionally, NIST traceable documentation for buffers, standards, calibration gases or reference materials will be maintained in a central file at the FEC.

For equipment maintained at the SESD laboratory and sent off-site for calibration certificates or other records of calibration will be maintained in the lab where the equipment is stored. Additionally, NIST traceable documentation for buffers, standards, calibration gases or reference materials will be maintained in a central file at the SESD laboratory with the equipment.

SESD will maintain records of actions taken to verify the quality of equipment whose properties could affect the quality of sampling, measurement and related activities. Examples would be thermometer, calibration standard or buffer verification. Thermometers used during field investigations will be verified by the SESD Analytical Support Branch (ASB) or EPA contract personnel utilizing the ASB Standard Operating Procedure for the Certification of Laboratory Thermometers, SOP ASB 100G. The SESD ASB is accredited by the National Environmental Laboratory Accreditation Conference.

Critical reference materials such as reagents and consumable materials that affect the quality of tests and/or calibrations will be verified according to the procedures described in the SESD Operating Procedure for Purchasing Services and Supplies, SESDPROC-015.

2.5.3 Equipment Performance Checks

New equipment will be calibrated or verified by qualified personnel to verify that it is adequate to perform its intended function, and calibrated before release for use in the field. Records of calibration or verification will be maintained in the equipment log books. If any equipment proves to be unsuitable for use, the BFEMs will document the issue, notify the purchasing official and return the deficient equipment to the vendor.

If equipment leaves the direct control of SESD for a period of time, the BFEMs will ensure that the function and, where necessary, the calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service.

2.6 Records

The BFEMs are responsible for maintaining a current equipment inventory list for their specific programs. The FQM will maintain a central, comprehensive list of all field measurement and sampling equipment subject to this procedure.

The BFEMs are responsible for creating equipment logs for all equipment in their specific program used for field measurement activities. Additionally, they are responsible for auditing and maintaining equipment maintenance, calibration and verification records in the equipment logs. These records must be made available to SESD personnel prior to their use for field measurement.

These records will include:

1. A copy of the Purchase Order, if available, or other record showing item received and date placed into service
2. Acceptance inspection record of the equipment (copy of signed invoice)
3. Maintenance, calibration and verification logs (including software if applicable)
4. Identification of individuals accessing maintenance, calibration and verification logs
5. Calibration certificates for manufacturers calibrations (if applicable)
6. Fundamental calibration or any other performance checks information
7. Calibration verification record (showing standards/reference materials used)
8. Calibration correction factors, if applicable
9. Records of handling, transportation and storage of equipment
10. Complaint log (if applicable)
11. A record of the most recent version of firmware or software for the equipment.

2.7 Equipment Sign-out/Sign-in

All measurement equipment will be signed-out prior to use in the field and signed-in following use in the field. This will be done using the SESD Field Equipment Tracking System or by using an equipment sign-out/sign-in form. Each form is specific to the type of measuring equipment being checked out and includes calibration or verification data, as well as, project name and project leader information.

FEC contract personnel will primarily be responsible for equipment sign-out and sign-in at the FEC. However, in their absence, it is the project leader's responsibility to sign out the measurement equipment. For measurement equipment that is not routinely handled or maintained by contract personnel or equipment stored at the SESD laboratory, the project leader or field investigator using the equipment is responsible for checking the equipment out and in.

Each equipment log will contain an equipment sign-out/sign-in form. Equipment defects or damage that is identified while in the field should be noted on the FEC Load-In Form (SESD FORM-011) when the equipment is signed in.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Measurement of Dissolved Oxygen**

Effective Date: January 8, 2014	**Number:** SESDPROC-106-R3

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-106-R3, *Field Measurement of Dissolved Oxygen*, replaces SESDPROC-106-R2 **General** Corrected any typographical, grammatical, and/or editorial errors. **Title Page** Changed Chief, Enforcement and Investigations Branch from Archie Lee to Danny France. Changed Chief, Ecological Assessment Branch from Bill Cosgrove to John Deatrick. Changed Field Quality Manager from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **Section 1.1**: In the last sentence, replaced "diffusion" with "reaeration." **Section 1.2:** Added the following statement: "Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use." **Section 2:** First Paragraph, Second Sentence: The phrase "taken to the field" was replaced with "utilized in the field". Third Paragraph: This paragraph, which discussed a 24 hour burn in period for Clarke Cell probes, was omitted. Fourth Paragraph: This paragraph, which discussed Winkler Titration, was omitted. **Section 3.1:** Converted part of the first paragraph into bulleted items. Omitted all language that referred to Winkler Titration. **Section 3.2:** Omitted all language that referred to Winkler Titration.	January 8, 2014
SESDPROC-106-R2, *Field Measurement of Dissolved Oxygen*, replaces SESDPROC-106-R1	February 12, 2010

SESDPROC-106-R1, *Field Measurement of Dissolved Oxygen*, replaces SESDPROC-106-R0	November 1, 2007
SESDPROC-106-R0, *Field Measurement of Dissolved Oxygen*, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes methods and considerations to be used and observed when conducting field measurements of dissolved oxygen in surface water, treated wastewater and in gas media for specific applications (e.g., reaeration measurement).

1.2 Scope/Application

On the occasion that SESD field investigators determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another method must be used to obtain a measurement of dissolved oxygen, the alternate procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Field Branches Quality Management Plan, SESDPLAN-001, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

Appropriate precautions should be observed when working in and around bodies of water and on boats. Be aware of fast flowing waters, waterway obstructions such as dams, and other vessels on the water.

2 Quality Control

All dissolved oxygen meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter is utilized in the field, it will be calibrated and verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

For in-situ measurements, an instrument warm-up period appropriate for that instrument should be provided. Consult manufacturer's documentation for appropriate warm-up time.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

Following instrument use, an end check should be performed using one of the techniques described in Section 3.2 to quantify potential instrument drift during use.

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Measurement of Dissolved Oxygen

3.1 General

Dissolved oxygen can be defined as the volume of oxygen contained in a volume of water. The solubility of oxygen in water is dependant on the water temperature, salinity and pressure.

- As the temperature of the water decreases, the solubility of oxygen increases.
- As salinity increases, the solubility of oxygen decreases.
- As the pressure decreases (altitude increases), the solubility of oxygen decreases.

Several methods for measurement of dissolved oxygen in water are available utilizing a variety of technologies.. When measuring dissolved oxygen for compliance with the National Pollutant Discharge Elimination System (NPDES) Program, only approved methods will be used. Approved methods can be found in the Code of Federal Regulations (CFR) 40 CFR Part 136.

3.1.1 Clark Cell Probes

Clark cell probes utilize an oxygen permeable membrane that covers an electrolytic cell which consists of a cathode and an anode. The anode acts as a reference electrode. After passing through the permeable membrane, the oxygen is reduced by an applied potential voltage that is referenced to the anode. The reduction current at the cathode is directly proportional to the partial pressure of oxygen in liquid, expressed as %-air saturation. The concentration of oxygen, in mg/l, is calculated based on the %-air saturation reading and the solubility of oxygen in water at the sample temperature.

In general, sample collection using a DO probe requires only lowering the probe into the sample media and recording or logging the results. The probe should be lowered gently to prevent damage to the membrane and gently turned when initially lowered to remove any attached air bubbles. If the instrument requires the use of a stirrer, the stirrer should be turned on before recording any readings. Prior to use, the instrument should be calibrated and any manufacturer specified warm-up period should be observed.

3.1.2 Luminescent Probes

Luminescent dissolved oxygen probes employ a light emitting diode (LED) to provide incident light, which excites the oxygen-sensitive luminescent-dye molecule substrate of the sensor. After dissipation of the excitation energy, longer-wavelength light is emitted (luminescence). The magnitude of steady-state

luminescence (intensity) is measured by the sensor and is inversely proportional to the dissolved oxygen concentration.

Sample collection with this type of probe should follow the sample procedures described in the second paragraph of Section 3.1.2 for Clark Cell probes.

3.2 Calibration

Many brands of instruments are commercially available for *in-situ* measurement of dissolved oxygen using Clark cell probes and luminescent probes. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate.

In general, calibrations should be conducted at temperatures and pressures as close as possible to those of the sample media for the most accurate measurements. Due to the sensitivity of dissolved oxygen measurements to changes in temperature, the temperature probe or thermistor should be verified using a NIST traceable thermometer prior to each calibration. Most dissolved oxygen meters utilize a one-point calibration which is generally performed using either water saturated air or air-saturated water. When using the water-saturated air method, the probes should be placed in a 100% relative humidity environment and the temperature and dissolved oxygen readings should be allowed to equilibrate. After equilibration, the meter should be set to read the appropriate dissolved oxygen concentration based on the temperature and barometric pressure.

When using air-saturated water for calibration, an aeration device such as an aquarium pump with a diffusion stone should be placed in a vessel containing tap water. The water in the vessel should be aerated for a minimum of one hour at a constant temperature. Saturation should be verified by placing the dissolved oxygen probe in the vessel and monitoring the temperature and dissolved oxygen readings for stabilization. Avoid placing the probe in the direct stream of air bubbles. Bubbles can accumulate on the probe surface and cause erroneous readings. Once the water is saturated, the temperature of the water and the barometric pressure can be used to determine the dissolved oxygen value. The meter can then be set to read that value.

3.3 Maintenance

Maintenance procedures vary depending on the technology utilized by each instrument and the manufacturer. The manufacturer's instruction manual should be consulted for instrument specific procedures. Following are some general guidelines for maintaining dissolved oxygen meters:

- Inspect probes for damage prior to use.

- For Clark cell probes, membranes and electrolyte solution should be changed prior to each study, when feasible.
- Battery voltages should be checked. For meters that will be deployed unattended, new or fully charged batteries should be used for each study.
- All calibration and maintenance procedures performed should be thoroughly documented.

3.4 Conducting Field Measurement of Dissolved Oxygen

Following are guidelines for conducting field measurements of dissolved oxygen:

- Except as described in specific operating procedures, dissolved oxygen measurements should if possible be conducted *in-situ*.
- When measuring DO at distinct points in the water column, the probe should be allowed to equilibrate at each location prior to recording the measurement.
- In water bodies with a great deal of flow, a weight may be attached to the probe guard or support cable to insure the probe is maintained at the proper depth.
- Insure that the measurement location is representative of conditions within the water body or reach. Avoid measurements directly below turbulent sections or still water unless these conditions represent most of the water body or reach.
- If DO measurements are conducted in saline water, the DO meter should either be capable of correcting for salinity or a separate instrument should be used to measure salinity so that the final DO measurements can be corrected.

3.5 Operational Check

A post-operation instrument verification check will be performed using one of the techniques described in Section 3.2 to quantify potential instrument drift during use. A verification check will be performed at the end of all measurements for a day or at the end of a deployment. The verification DO concentration will be measured and recorded in the field logbook prior to any instrument adjustment.

It may be appropriate to check the calibration of a DO meter periodically during the course of a day's measurements when conducting individual measurements rather than deploying an instrument. When this is done, it should be noted in the field logbook. The calibration of meters checked throughout the day maybe adjusted if drift is occurring.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Equipment Cleaning and Decontamination**

Effective Date: December 20, 2011	**Number:** SESDPROC-205-R2

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-205-R2, *Field Equipment Cleaning and Decontamination*, replaces SESDPROC-205-R1. **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Changed the Enforcement and Investigations Branch Chief from Antonio Quinones to Archie Lee. Changed the Field Quality Manager form Laura Ackerman to Bobby Lewis. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted reference to the H: drive. Changed the Field Quality Manager to the Document Control Coordinator. **Section 1.4:** In definitions (third bullet), replaced "soap" with "detergent." Changed all references in document from "soap" to "detergent." **Section 1.5:** Updated references. **Section 2.1:** On the 2nd paragraph, removed the last two sentences and replaced with new language. The new language clarifies that any deviations from the standard decon procedures that need to be made during the course of the field investigation will be documented in the field logbook. **Section 3.5**: Revised to clarify that it is only necessary to decontaminate the wetted portion of the sounder or tape. **Section 3.6**: Rewritten to reflect updated RediFlo2® internal cleaning procedures. **Section 3.7:** Added Section 3.7.5*, Field Decontamination Procedure for Direct Push Technology (DPT) Equipment.*	December 20, 2011

SESDPROC-205-R1, *Field Equipment Cleaning and Decontamination*, replaces SESDPROC-205-R0. **General** Corrected any typographical, grammatical and/or editorial errors. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.5** Alphabetized and revised the referencing style for consistency. **Section 1.6.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.	November 1, 2007
SESDPROC-205-R0, *Field Equipment Cleaning and Decontamination*, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when cleaning and decontaminating sampling equipment during the course of field investigations.

1.2 Scope/Application

The procedures contained in this document are to be followed when field cleaning sampling equipment, for both re-use in the field, as well as used equipment being returned to the Field Equipment Center (FEC). On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that other procedures must be used to clean or decontaminate sampling equipment at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

- Decontamination: The process of cleaning dirty sampling equipment to the degree to which it can be re-used, with appropriate QA/QC, in the field.

- Deionized water: Tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard inductively coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Deionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for deionized water.

- Detergent: A standard brand of phosphate-free laboratory detergent, such as Luminox®.

- Drilling Equipment: All power equipment used to collect surface and sub-surface soil samples or install wells. For purposes of this procedure, direct push is also included in this definition.

- Field Cleaning: The process of cleaning dirty sampling equipment such that it can be returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

- Organic-free water: Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

- Tap water: Water from any potable water supply. Deionized water or organic-free water may be substituted for tap water.

1.5 References

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.6 General Precautions

1.6.1 Safety

Proper safety precautions must be observed when field cleaning or decontaminating dirty sampling equipment. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant

requirements, as appropriate. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- When conducting field cleaning or decontamination using laboratory detergent, safety glasses with splash shields or goggles, and latex gloves will be worn.

- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

1.6.2 Procedural Precaution

Prior to mobilization to a site, the expected types of contamination should be evaluated to determine if the field cleaning and decontamination activities will generate rinsates and other waste waters that might be considered RCRA hazardous waste or may require special handling.

2 Introduction to Field Equipment Cleaning and Decontamination

2.1 General

The procedures outlined in this document are intended for use by field investigators for cleaning and decontaminating sampling and other equipment in the field. These procedures should be followed in order that equipment is returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for the Data Quality Objectives (DQOs) of the study or investigation. If deviations from these procedures need to be made during the course of the field investigation, they will be documented in the field logbook along with a description of the circumstances requiring the use of the variant procedure.

Cleaning procedures for use at the Field Equipment Center (FEC) are found in SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC (SESDPROC-206).

2.2 Handling Practices and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Detergent must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.

- Tap water may be kept in tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.

- Deionized water must be stored in clean, glass or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.

- Organic-free water must be stored in clean glass or Teflon® containers prior to use. It may be applied using Teflon® squeeze bottles, or with the portable system.

2.3 Disposal of Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW); including used wash water and rinse water are in SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202).

2.4 Sample Collection Equipment Contaminated with Concentrated Materials

Equipment used to collect samples of concentrated materials from investigation sites must be field cleaned before returning from the study. At a minimum, this should consist of washing with detergent and rinsing with tap water. When the above procedure cannot be followed, the following options are acceptable:

1. Leave with facility for proper disposal;

2. If possible, containerize, seal and secure the equipment and leave on-site for later disposal;

3. Containerize, bag or seal the equipment so that no odor is detected and return to the SESD.

It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.5 Sample Collection Equipment Contaminated with Environmental Media

Equipment used to collect samples of environmental media from investigation sites should be field cleaned before returning from the study. Based on the condition of the sampling equipment, one or more of the following options must be used for field cleaning:

1. Wipe the equipment clean;

2. Water-rinse the equipment;

3. Wash the equipment in detergent and water followed by a tap water rinse.

4. For grossly contaminated equipment, the procedures set forth in Section 2.4 must be followed.

Under extenuating circumstances such as facility limitations, regulatory limitations, or during residential sampling investigations where field cleaning operations are not feasible, equipment can be containerized, bagged or sealed so that no odor is detected and returned to the FEC without being field cleaned. If possible, FEC personnel should be notified that equipment will be returned without being field cleaned. It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.6 Handling of Decontaminated Equipment

After decontamination, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the decontamination area to prevent re-contamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

3 Field Equipment Decontamination Procedures

3.1 General

Sufficient equipment should be transported to the field so that an entire study can be conducted without the need for decontamination. When equipment must be decontaminated in the field, the following procedures are to be utilized.

3.2 Specifications for Decontamination Pads

Decontamination pads constructed for field cleaning of sampling and drilling equipment should meet the following minimum specifications:

- The pad should be constructed in an area known or believed to be free of surface contamination.
- The pad should not leak.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
- Water should be removed from the decontamination pad frequently.
- A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for proper handling and disposal of these materials. If the decontamination pad has leaked excessively, soil sampling may be required.

3.3 "Classical Parameter" Sampling Equipment

"Classical Parameters" are analyses such as oxygen demand, nutrients, certain inorganic compounds, sulfide, flow measurements, etc. For routine operations involving classical parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample water or tap water between sampling locations as appropriate.

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment may be cleaned with tap water between measuring locations, if necessary.

Note: The procedures described in Section 3.3 are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.

3.4 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

For samples undergoing trace organic or inorganic constituent analyses, the following procedures are to be used for all sampling equipment or components of equipment that come in contact with the sample:

1. Clean with tap water and Luminox® detergent using a brush, if necessary, to remove particulate matter and surface films. Equipment may be steam cleaned (Luminox® detergent and high pressure hot water) as an alternative to brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items should not be steam cleaned.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with organic-free water and place on a clean foil-wrapped surface to air-dry.

4. All equipment must be wrapped with foil. If the equipment is to be stored overnight before it is wrapped in foil, it should be covered and secured with clean, unused plastic sheeting.

3.5 Well Sounders or Tapes

The following procedures are recommended for decontaminating well sounders (water level indicators) and tapes. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the sounder or tape.

1. Wash with detergent and tap water.

2. Rinse with tap water.

3. Rinse with deionized water.

3.6 Redi-Flo2® Pump

CAUTION – Do not wet the controller. Always disconnect power from the pump when handling the pump body.

The Redi-Flo2® pump and any associated connected hardware (e.g., check valve) should be decontaminated between each monitoring well. The following procedures are required, depending on whether the pump is used solely for purging or used for purging and sampling.

3.6.1 Purge Only (Pump and Wetted Portion of Tubing or Hose)

1. Disconnect power and wash exterior of pump and wetted portion of the power lead and tubing or hose with detergent and water solution.

2. Rinse with tap water.

3. Final rinse with deionized water.

4. Place pump and reel in a clean plastic bag and keep tubing or hose contained in clean plastic or galvanized tub between uses.

3.6.2 Purge And Sample

Grundfos Redi-Flo2® pumps are extensively decontaminated and tested at the FEC to prevent contamination from being transmitted between sites. The relevant sections of SESDPROC-206, *Field Equipment Cleaning and Decontamination at the FEC,* should be implemented in the field where a high risk of cross-contamination exists, such as where NAPL or high-concentration contaminants occur. In most cases, the abbreviated cleaning procedure described below will suffice.

1. Disconnect and discard the previously used sample tubing from the pump. Remove the check valve and tubing adapters and clean separately (See Section 3.6.3 for check valve). Wash the pump exterior with detergent and water.

2. Prepare and fill three containers with decontamination solutions, consisting of Container #1, a tap water/detergent washing solution; Container #2, a tap water rinsing solution; and Container #3, a deionized or organic-free water final rinsing solution. Choice of detergent and final rinsing solution for all steps in this procedure is dependent upon project objectives (analytes and compounds of interest). The containers should be large enough to hold the pump and one to two liters of solution.

3. Place the pump in Container #1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.

4. Place the pump in Container #2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.

5. Place the pump in Container #3. Turn the pump on and circulate deionized or organic-free water through the pump and then turn the pump off.

6. Disconnect power and remove pump from Container #3. Rinse exterior and interior of pump with fresh deionized or organic-free water.

7. Decontaminate the power lead by washing with detergent and water, followed by tap water and deionized water rinses. This step may be performed before washing the pump if desired.

8. Reassemble check valve and tubing adapters to pump. ALWAYS use Teflon® tape to prevent galling of threads. Firm hand-tightening of fittings or light wrench torque is generally adequate.

9. Place the pump and reel in a clean plastic bag.

3.6.3 Redi-Flo2® Ball Check Valve

1. Remove the ball check valve from the pump head. Check for wear and/or corrosion, and replace as needed. During decontamination check for free-flow in forward direction and blocking of flow in reverse direction.

2. Using a brush, scrub all components with detergent and tap water.

3. Rinse with deionized water.

4. Replace the ball check valve to the Redi-Flo2® pump head.

3.7 Downhole Drilling Equipment

These procedures are to be used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

3.7.1 Introduction

Cleaning and decontamination of all equipment should occur at a designated area (decontamination pad) on the site. The decontamination pad should meet the specifications of Section 3.2 of this procedure.

Tap water brought on the site for drilling and cleaning purposes should be contained in a pre-cleaned tank.

A steam cleaner and/or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam (200° F plus), with a detergent compartment, should be obtained.

3.7.2 Preliminary Cleaning and Inspection

Drilling equipment should be clean of any contaminants that may have been transported from off-site to minimize the potential for cross-contamination. The drilling equipment should not serve as a source of contaminants. Associated drilling and decontamination equipment, well construction materials, and equipment handling procedures should meet these minimum specified criteria:

- All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (detergent and high pressure hot water), or wire brushing. Sandblasting should be performed <u>prior to arrival</u> on site, or well away from the decontamination pad and areas to be sampled.

- Any portion of the drilling equipment that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent

and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other sites before being brought on site.

- Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can provide materials without the printing and/or writing if specified when ordered. Items that cannot be cleaned are not acceptable and should be discarded.

- Equipment associated with the drilling and sampling activities should be inspected to insure that all oils, greases, hydraulic fluids, etc., have been removed, and all seals and gaskets are intact with no fluid leaks.

3.7.3 Drill Rig Field Cleaning Procedure

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) between boreholes.

3.7.4 Field Decontamination Procedure for Drilling Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment. This procedure does <u>not</u> apply to well casings, well screens, or split-spoon samplers used to obtain samples for chemical analyses, which should be decontaminated as outlined in Section 3.4 of this procedure.

1. Wash with tap water and detergent, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with detergent) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.

2. Rinse thoroughly with tap water.

3. Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

3.7.5 Field Decontamination Procedure for Direct Push Technology (DPT) Equipment

1. Certain specific procedures for the decontamination of DPT tools are described in the various sampling procedures, but the following general guidelines apply:

2. Prior to return to the Field Equipment Center, all threaded tool joints should be broken apart and the equipment cleaned per the provisions of *Section 2.5, Sample Collection Equipment Contaminated with Environmental Media* of this procedure.

3. Equipment that contacts the sample media and is cleaned in the field for reuse should be cleaned per the provisions of *Section 3.4, Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds* of this procedure. This would include piston sampler points and shoes, screen point sampler screens and sheaths, and the drive rods when used for groundwater sampling.

4. Equipment that does not directly contact the sample media and is cleaned in the field for reuse can generally be cleaned per the provisions of Section *3.7.4, Field Decontamination Procedure for Drilling Equipment* of this procedure.

U.S. Environmental Protection Agency
Region 4, Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Measurement of Oxidation-Reduction Potential (ORP)**

Effective Date: January 29, 2013	**Number:** SESDPROC-113-R1

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Revision History

This Table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-113-R1, *Field Measurement of Oxidation-Reduction Potential (ORP)*, replaces SESDPROC-013-R0 **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the EIB Chief from Archie Lee to Danny France, and the Field Quality Manager from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 2.2:** Added new paragraph #7 and new Figure 4. Renumbered subsequent Figures. In paragraph #8, deleted the following sentence: "In multi parameter sondes, the reference electrode is typically shared by the ORP and pH measuring systems." In paragraph #10, 2nd sentence, updated the reference from Figure 3 to Table 1. In addition, updated numbers in the "example" equation. In paragraph #11, 1st sentence, replaced +560 mV with +544 mV. Added new paragraph #12 and new Figure 6. **Section 3.2:** **A**dded new paragraph #5 describing operational checks in the event of potential disturbances. In paragraph #8, added the following at the end of the 1st sentence: "or handled as directed by the SHEMP."	January 29, 2013
SESDPROC-113-R0, *Field Measurement of Oxidation-Reduction Potential (ORP)*, Original Issue	August 7, 2009

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1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field Oxidation-Reduction Potential (ORP) measurements in aqueous environmental media, including groundwater, surface water and certain wastewater. The measurement of soil ORP is a non-standard measurement and procedures should be developed on a project-specific basis.

1.2 Scope/Application

This document describes procedures generic to all ORP measurement methods to be used by Science and Ecosystem Support Division (SESD) field personnel when collecting and handling samples in the field. On the occasion SESD personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain an ORP measurement, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Faulkner, S.P., W.H. Patrick, Jr., and R.P. Gambrell. 1989. Field techniques for measuring wetland soil parameters. Soil Sci. Soc. Am. J. 53:883-890.

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1.5 General Considerations

1.5.1 Safety

Proper safety precautions must be observed when verifying or calibrating instruments for measurement of Oxidation-Reduction Potential. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual (most recent version) and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional.

Reagents commonly used in the preparation of ORP calibration standards are toxic and require care when handling. When using this procedure, avoid exposure to these materials through the use of protective clothing, eye wear and gloves. Safety precautions when handling and preparing verification solutions should include gloves and eyewear to prevent dermal and eye contact, and a mask to avoid inhaling dust particles when handling dry materials. Vigorous flushing should be used if the reagents or solutions come in contact with skin or eyes. Following is specific information on commonly used solutions. The application of the solutions is described in detail in Section 3.1, Standard Solutions, of this procedure.

- Quinhydrone (CAS# 106-34-3) is a skin and respiratory irritant and is poisonous if ingested. Safety precautions when handling quinhydrone should include gloves to prevent dermal contact and a mask to avoid inhaling dust particles when mixing dry material to prepare calibration standards. Vigorous flushing should be used if

concentrated material comes in contact with skin or eyes.

- Zobell's solution is also an irritant and toxic if ingested. The same handling precautions apply when mixing and using Zobell's solution as when using quinhydrone. Zobell's reacts with acid to form harmful byproducts, including hydrocyanide gas.

- Light's solution contains ferro- and ferric-cyanide compounds in sulfuric acid. The components are toxic and burns are possible from contact with this solution.

- Potassium iodide solutions have lower toxicity than most calibration solution options. General ingestion, skin contact, and eye contact precautions apply.

Unused quinhydrone, Zobell's, Light's or other calibration reagents and solutions should be returned to SESD for disposal in accordance with the SESD Safety, Health, and Environmental Management Plan (SHEMP).

1.5.2 Records

Documentation of field activities is done in a bound logbook. All records, including a unique, traceable identifier for the instrument, should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010, most recent version) and the SESD Operating Procedure for Equipment Inventory and Management, (SESDPROC-108, most recent version).

All field ORP measurements pertinent to the sampling event should be recorded in the field logbook for the event as outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010, most recent version), or managed electronically with appropriate backups as described in SESD Operating Procedure for Control of Records (SESDPROC-002, most recent version).

1.5.3 Shipping

Shipped material shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

2 Background

2.1 General

Oxidation is the process of liberating electrons or gaining oxygen. Examples of oxidation include conversion of elemental iron to rust, elemental sulfur to sulfate, and elemental hydrogen to water (Pankow 1991). Reduction is the process of gaining electrons resulting in the charge on some atomic unit in the species to be reduced. Oxidation-reduction potential (ORP) or redox potential (hereafter, referred to as redox) is a measure of the intensity or activity of an aqueous environment or soil to mediate reactions of important elements in biological systems (e.g., O, N, Mn, Fe, S, and C) and other metallic elements.

Considerable confusion arises on the use of the terms oxidation and reduction as they apply to the media under study. The following introduction reproduced from an online 'Wikipedia' article on the topic lucidly explains their relationship in ORP measurement:

> **Reduction potential** (also known as **redox potential**, **oxidation / reduction potential** or **ORP**) is the tendency of a chemical species to acquire electrons and thereby be reduced. Each species has its own intrinsic reduction potential; the more positive the potential, the greater the species' affinity for electrons and tendency to be reduced.
>
> In aqueous solutions, the reduction potential is the tendency of the solution to either gain or lose electrons when it is subject to change by introduction of a new species. A solution with a higher (more positive) reduction potential than the new species will have a tendency to gain electrons from the new species (i.e. to be reduced by oxidizing the new species) and a solution with a lower (more negative) reduction potential will have a tendency to lose electrons to the new species (i.e. to be oxidized by reducing the new species). Just as the transfer of hydrogen ions between chemical species determines the pH of an aqueous solution, the transfer of electrons between chemical species determines the reduction potential of an aqueous solution. Like pH, the reduction potential represents an intensity factor. It does not characterize the capacity of the system for oxidation or reduction, in much the same way that pH does not characterize the buffering capacity.

In short, a numerically positive redox potential or ORP represents an environment conducive to the oxidation of an introduced substance by reduction of the original media.

2.2 Instrumentation

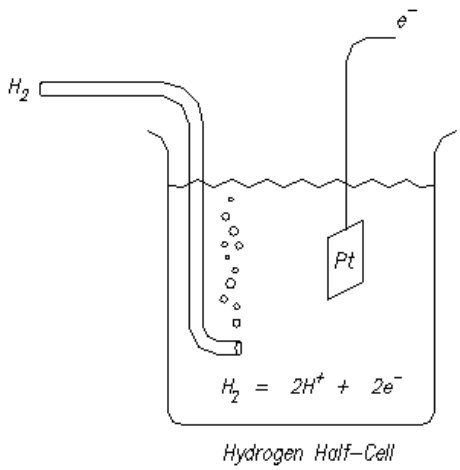
ORP measurement systems are a practical implementation of electrochemical cells, which use metal electrodes in a solution to generate an electric current or voltage. If a platinum electrode is immersed in water with hydrogen bubbled into the solution, the H^2 is oxidized as follows:

$$H^2 = 2H^+ + 2e^-$$

In the electrochemical half-cell illustrated below in Fig.1, hydrogen gas oxidizes to hydrogen ions and free electrons, comprising an oxidation-reduction couple. This couple reaches an

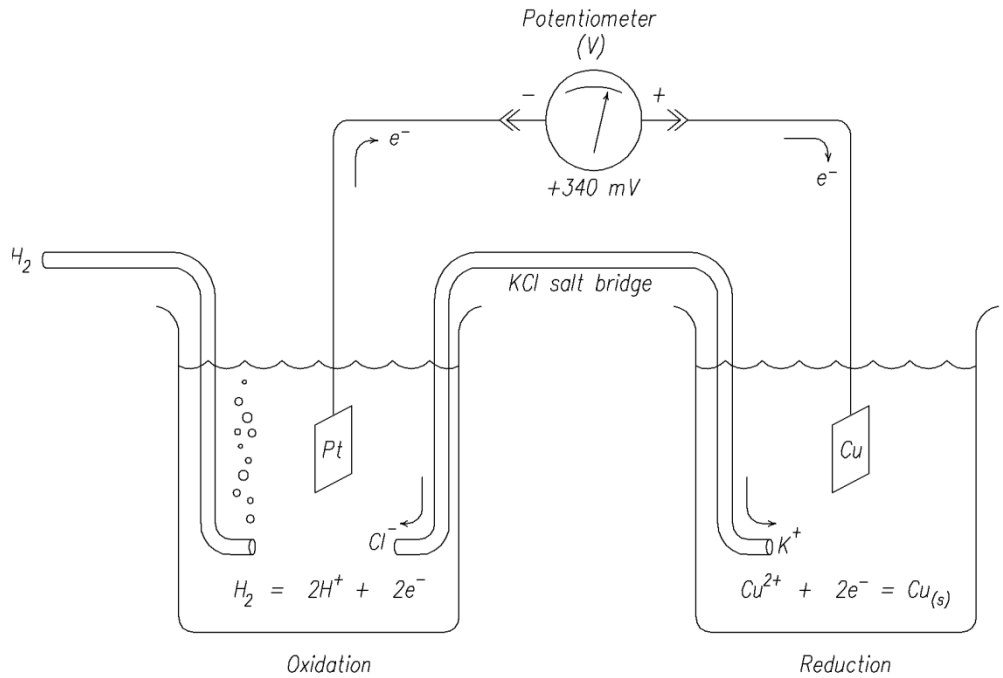
equilibrium state that maintains the reference potential of the electrode. The electric potential develops on the wire connected to the platinum electrode, but is difficult to measure in practice in the isolated half-cell. However, when used in a complete electrochemical cell, the cell illustrated is used as a reference to measure other half-cells against, and is called a Standard Hydrogen Electrode (SHE).

Figure 1



If, as shown in Figure 2, a SHE is connected with a salt bridge to a second half-cell in which a reduction reaction is taking place, the electric potential between the two cells can be measured. In the case shown, the potential of the right cell will be +0.34 Volts in reference to the standard hydrogen electrode on the left. This would be represented as an Oxidation Reduction Potential (ORP) of +340mV on the hydrogen scale, or simply as Eh = +340mV.

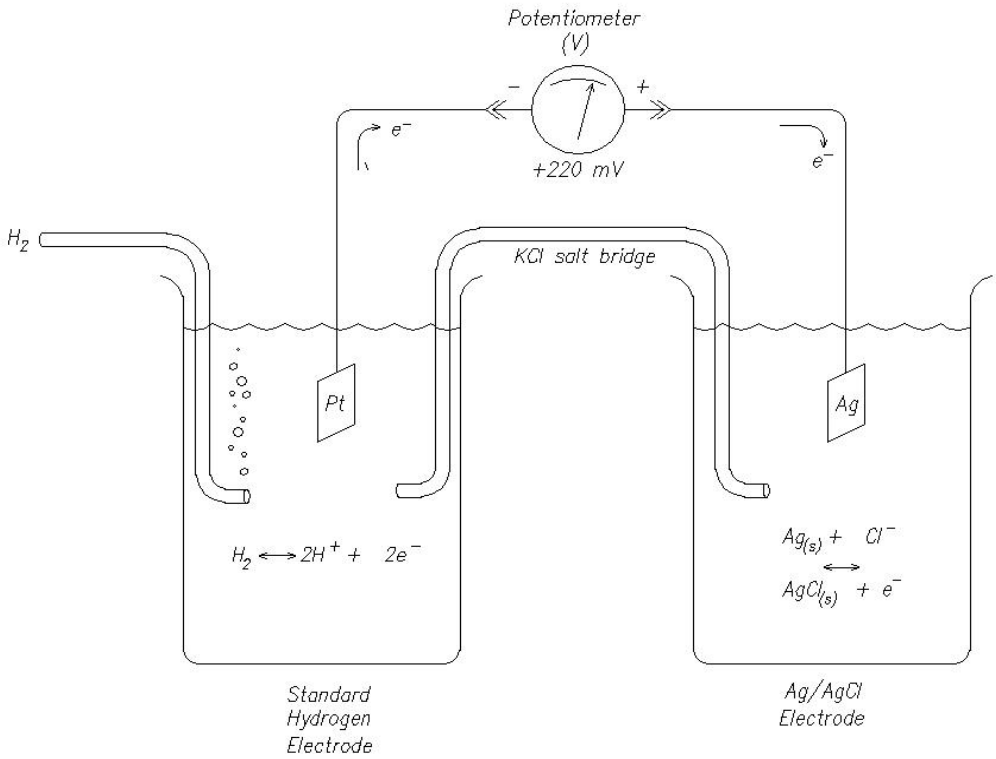
Figure 2



In field practice, the hydrogen electrode is difficult to reproduce. To conduct field measurements, a reference electrode is needed that is simple to maintain and will generate a potential that can be referenced to the standard hydrogen electrode. These requirements are met by the Saturated Calomel Electrode (SCE) and the Silver/Silver Chloride Electrode (SSCE - the SSCE is also commonly identified as an Ag/AgCl electrode). The SCE contains a small amount of elemental mercury, and while useful for certain applications, would rarely be used at SESD. The SSCE or Ag/AgCl electrode is generally used as the reference cell in SESD instrumentation.

In Figure 3 below, a SHE is connected to an Ag/AgCl electrode. In this example of an electrochemical cell, both cells reach an equilibrium potential. At that equilibrium state, the potential of the Ag/AgCl cell is 220mV more positive than the standard hydrogen electrode.

Figure 3



This half-cell potential of the Ag/AgCl electrode in reference to the SHE is used to convert measurements taken with an Ag/AgCl reference back to the hydrogen scale. While the laboratory Ag/AgCl half-cell shown has a potential of +220mV, practical reference cells have varying potentials based on temperature and filling solutions as shown in Table 1 below.

Table 1

Half-cell Potential of Ag/AgCl reference electrode

derived from USGS NFM, Table 6.5.2 (9/2005)

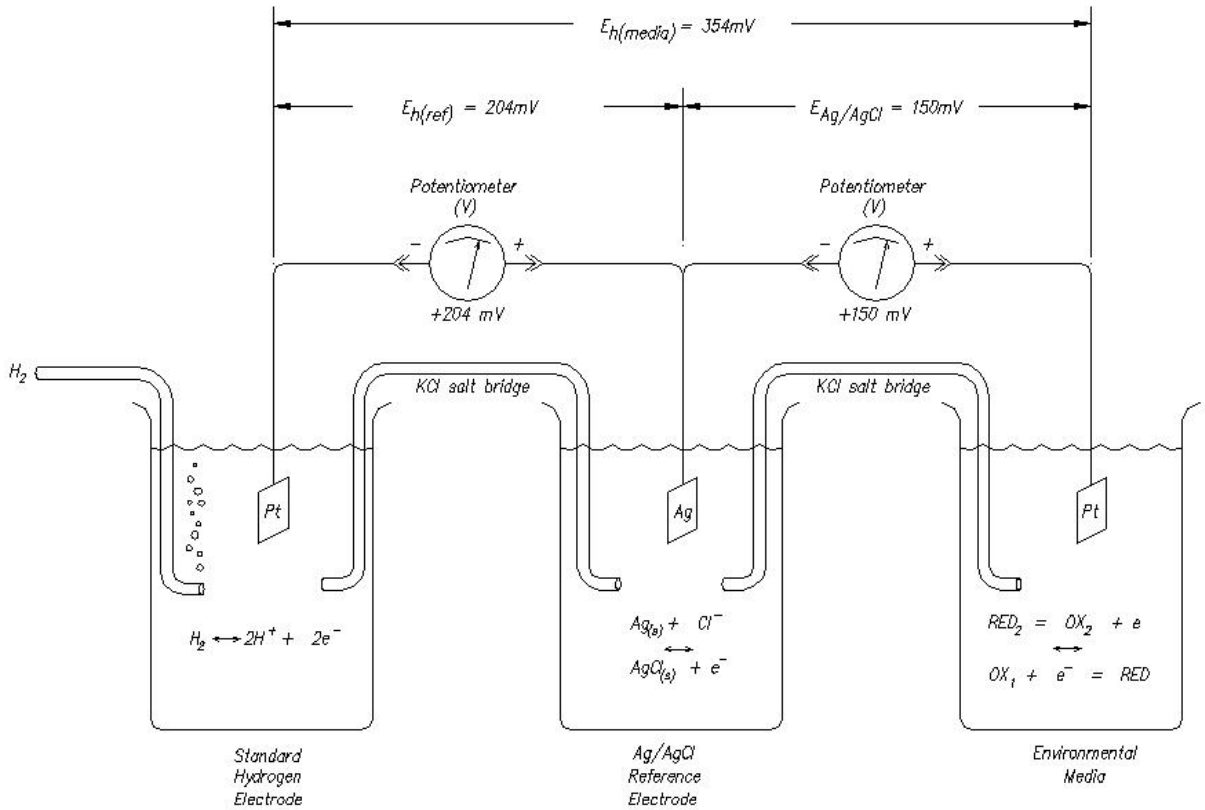
	Molarity of KCl filling solution			
T(°C)	3M	3.3M*	3.5M	Sat/4M
10	220	217	215	214
15	216	214	212	209
20	213	210	208	204
25	209	207	205	199
30	205	203	201	194
35	202	199	197	189
40	198	195	193	184

*interpolated value

Note: YSI sondes and Thermo electrodes typically use 4M KCl filling solutions. Eureka sondes typically use 3.3M KCl filling solutions.

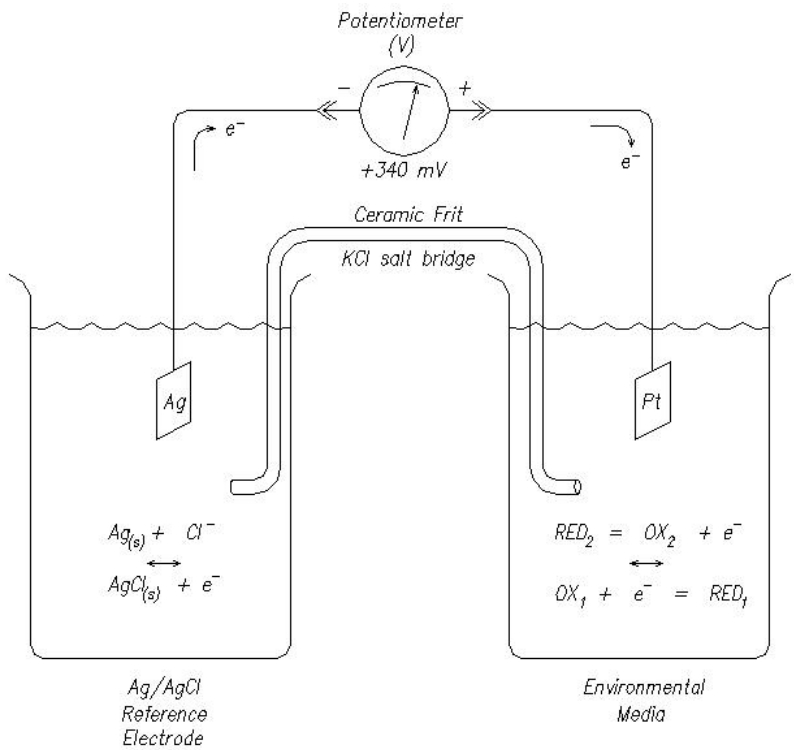
In Figure 4, below, the relationship between a hydrogen electrode, a reference electrode, and a platinum sensing electrode in an arbitrary media is shown. In this case, the ORP of the media in reference to the silver/silver chloride electrode is 150mV. To obtain Eh, the potential of the reference electrode in relation to a hydrogen electrode is added to the potential of the sensing electrode in relation to the reference electrode. In practice, the potential of the reference electrode in relation to a hydrogen electrode is not measured, but obtained from Table 1 above.

Figure 4



In Figure 5 below, a field instrument is represented as separate electrochemical cells. The Ag/AgCl reference electrode uses a ceramic frit or other means to provide the essential salt bridge to the environmental media. The platinum sensing electrode is immersed in the environmental media and connected internally in the instrument to measure the potential (voltage) between the two electrodes.

Figure 5



In this illustration, the ORP is measured as 340 mV. This measurement is made in reference to the Ag/AgCl reference electrode and would be reported as such, or as $E_{Ag/AgCl}$ = 340mV.

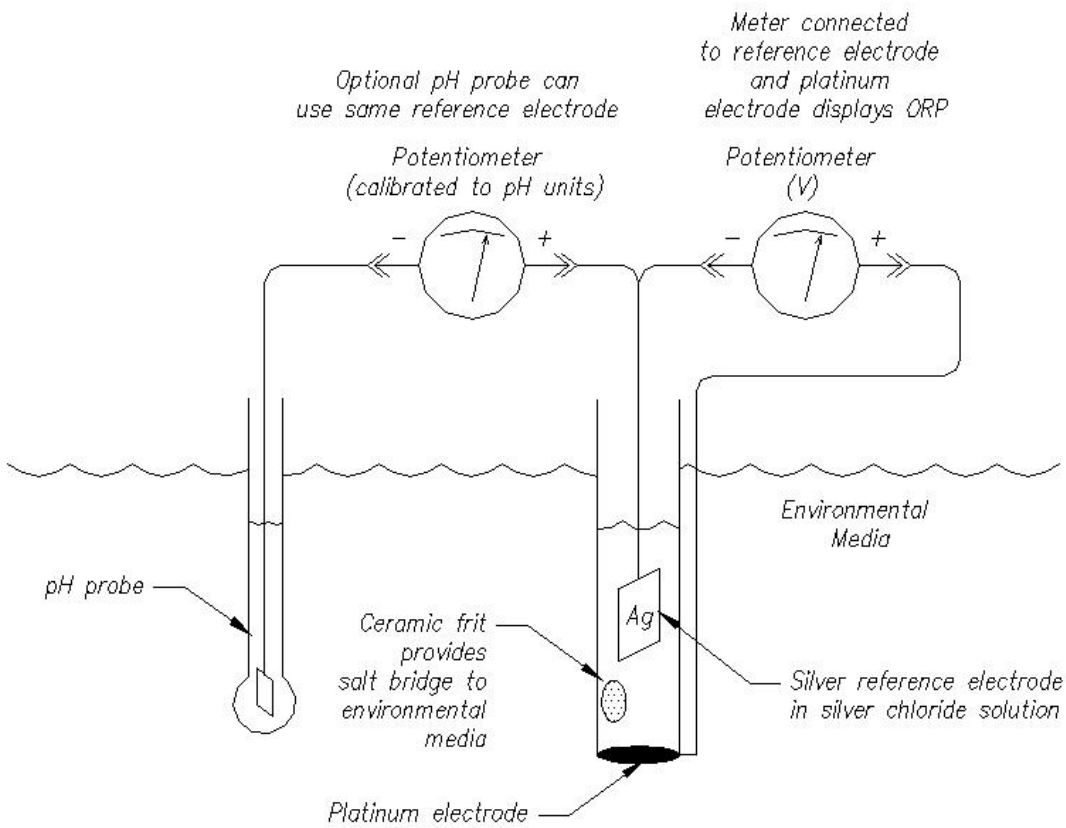
In some cases it will be desirable to report the reading on the hydrogen scale, or Eh. To do so, the potential of the reference electrode against the SHE, obtained from Table 1, is added to $E_{Ag/AgCl}$. For our example:

	340 mv	Measured ORP ($E_{Ag/AgCl}$) of sample
+	204 mV	Eh of Ag/AgCl electrode (ORP of Ag/AgCl electrode referenced to SHE)
	544 mV	Eh of sample

Both the +340 mV field reading and the corrected +544 mV Eh can properly be referred to as ORP results. It is only through specifying the reference scale that the ambiguity can be eliminated.

In Figure 6, below, the theoretical cells shown above have been configured as a practical field instrument. The salt bridge is commonly provided by a ceramic frit connecting the environmental media to the reference electrode. In multi-parameter sondes, the pH probe commonly uses the same reference electrode as the ORP probe.

Figure 6



2.3 Redox Chemistry

In acid-base chemistry, the pH of a system is defined as the negative logarithm of the hydrogen ion activity (simplified in practice to the hydrogen ion concentration):

$$pH = -\log \{H^+\}$$

Similarly, Pankow (1991) described the negative logarithm of the electron activity (pe) as the master variable for describing the equilibrium position for all redox couples in a given system:

$$pe \;/\; -\log \{e^-\}$$

It can be shown (Pankow) that pe is related to Eh by

$$Eh = pe*(2.303*R*T)/F$$

Where:

R = gas constant = $8.314\ J\ K^{-1}\ mol^{-1}$
T = temperature, ^{o}K
F = Faraday constant = $96.485*10^{3}\ C\ mol^{-1}$

At 25°C (298°K) this simplifies to

$$E_H = pe * 0.05916$$

And

$$pe = E_H / 0.05916$$

According to Faulkner et al. (1989) redox is a quantitative measure of electron availability and is indicative of the intensity of oxidation or reduction in both chemical and biological systems. When based on a hydrogen scale, redox (E_H) is derived from the Nernst Equation (Stumm and Morgan 1981):

$$E_H = E_H^{o} + 2.3\ H\ (R\ H\ T)/nF\ H \log(J_i\ \{ox\}^{ni} / J_j\ \{red\}^{nj})$$

Where:

E_H^{o} = potential of reference, mV
R = gas constant = $81.987\ cal\ deg^{-1}\ mole^{-1}$
T = temperature, ^{o}K
n = number of moles of electrons transferred
F = Faraday constant = 23.061 cal/mole-mv
{ox} and {red} = activity of the oxidants and reductants, respectively

2.4 Applications

When interpreted properly, redox combined with other conventional water quality parameters is useful in developing a more complete understanding of water chemistry. Several applications of redox are identified below:

1. Redox could be viewed as an extension of the oxygen scale. In this model, the DO probe spans the aerobic scale and the redox probe extends that scale to measure anaerobic conditions. Inferences to geochemistry and chemical speciation can be made from the oxidative state of the system. Application to metal sequestration, metal-iron, -sulfide, -methane complexation, and the subsequent bioaccumulation potential is possible.
2. Redox can be used to identify anaerobiosis at or near the water column and sediment interface in streams, lakes, and estuaries.
3. Redox may be useful in determination of stream jurisdiction and wetland delineation in that it can indicate conditions of soil saturation.

4. Based on redox, a pe (or EH) vs. pH stability diagram can be developed to aid in nutrient exchange studies including the timing, release, and partitioning of important water and sediment quality pollutants such as nitrogen and phosphorus species. Most importantly, redox can be used to address error associated with chamber-effect during closed chamber measurements of the water-sediment interface. Redox probes placed inside the contact chamber and inserted approximately ten centimeters into the underlying sediment can be used to monitor changes in sediment redox caused by the chamber, and steps can be taken to reduce chamber-effect.
5. Redox may be useful in establishing water and sediment quality standards applicable to wetlands.
6. Redox is used to assess the potential of a groundwater system to support various in situ reactions with contaminants, such as reductive dechlorination of chlorinated solvents.
7. Redox can provide a useful indicator of conditions that might compromise the performance of Clark-type dissolved oxygen (DO) probes. In general, anaerobic conditions occur at a redox range of +150 mV to +300 mV (pH-dependent and adjusted to hydrogen reference electrode). When redox drops below this level, DO measurements as determined with a Clarke-type probe are highly suspect as the semi-permeable membrane does not discriminate between partial O_2 and sulfides. Consequently, the meter may be reading sulfides.

2.5 Limitations

In most environmental media, redox reactions will not reach equilibrium due to low concentrations or multiple redox species. Consequently, redox measurements can generally be considered semi-quantitative in environmental media, unless certain conditions exist.

The USGS in the Interferences and Limitations Section 6.5.3A of their National Field Manual succinctly describe some of the issues encountered in the application of ORP measurements. This section is reproduced here, unedited:

> 6.5.3.A INTERFERENCES AND LIMITATIONS
>
> *Measurements should not be carried out without an awareness of the interferences and limitations inherent in the method.*
>
> • *Organic matter and sulfide may cause contamination of the electrode surface, salt bridge, or internal electrolyte, which can cause drift or erratic performance when reference electrodes are used (American Public Health Association and others, 2001).*
>
> • *Hydrogen sulfide can produce a coating on the platinum electrode that interferes with the measurement if the electrode is left in sulfide-rich water for several hours (Whitfield, 1974; Sato, 1960).*
>
> • *The platinum single and combination redox electrodes may yield unstable readings in solutions containing chromium, uranium, vanadium, or titanium ions and other ions that*

are stronger reducing agents than hydrogen or platinum (Orion Research Instruction Manual, written commun., 1991).

- *Do not insert redox electrodes into iron-rich waters directly after the electrode(s) contact ZoBell's. An insoluble blue precipitate coats the electrode surface because of an immediate reaction between ferro- and ferricyanide ions in ZoBell's with ferrous and ferric ions in the sample water, causing erratic readings.*

Many elements with more than one oxidation state do not exhibit reversible behavior at the platinum electrode surface and some systems will give mixed potentials, depending on the presence of several different couples (Barcelona and others, 1989; Bricker, 1982, p. 59–65; Stumm and Morgan, 1981, p. 490–495; Bricker, 1965, p. 65). Methane, bicarbonate, nitrogen gas, sulfate, and dissolved oxygen generally are not in equilibrium with platinum electrodes (Berner, 1981).

TECHNICAL NOTE:

Misconceptions regarding the analogy between Eh (pe) and pH as master variables and limitations on the interpretation of Eh measurements are explained in Hostettler (1984), Lindberg and Runnells (1984), Thorstenson (1984), and Berner (1981). To summarize:

(1) Hydrated electrons do not exist in meaningful concentrations in most aqueous systems—in contrast, pH represents real activities of hydrated protons. Eh may be expressed as pe (the negative logarithm of the electron activity), but conversion to pe offers no advantage when dealing with measured potentials.

(2) Do not assume that redox species coexist in equilibrium. Many situations have been documented in which dissolved oxygen coexists with hydrogen sulfide, methane, and ferrous iron.

- *The practicality of Eh measurements is limited to iron in acidic mine waters and sulfide in waters undergoing sulfate reduction.*

- *Other redox species are not sufficiently electroactive to establish an equilibrium potential at the surface of the conducting electrode.*

(3) A single redox potential cannot be assigned to a disequilibrium system, nor can it be assigned to a water sample without specifying the particular redox species to which it refers. Different redox elements (iron, manganese, sulfur, selenium, arsenic) tend not to reach overall equilibrium in most natural water systems; therefore, a single Eh measurement generally does not represent the system.

3 Methodology

3.1 Standard Solutions

Care should be taken not to contaminate standards and samples and to verify the expiration date of all standards prior to use. All meters should be verified or calibrated according to the manufacturer's procedures.

Standard solutions for calibration and verification should be selected to meet project requirements. SESD generally maintains a stock of Zobell's solution suitable for most projects. The characteristics and use of the common standard solutions are described below.

- Zobell's solution contains potassium ferri- and ferro- cyanide compounds. The solution is available as prepared solutions or premeasured reagents for mixing by the user. Zobell's has moderate toxicity but will react with acid to form harmful byproducts, including hydrocyanide gas. It has a shelf life ranging from several days to several months depending on the manufacturer. Stock and working solutions of Zobell's should be stored in dark bottles due to its light sensitivity.

- Quinhydrone solutions are mixed at the time of use by adding quinhydrone to pH 4 or pH 7 buffers. At 25°C, the E_h of quinhydrone pH 4 and pH 7 verification solutions are 462mV and 285mV respectively. An advantage of quinhydrone solutions is that they offer a span of calibration points that may be appropriate for particular applications. Quinhydrone is a lightly 'poised' solution in that it offers less driving force towards the calibration point: a compromised instrument is more likely to be revealed in a quinhydrone calibration. A quinhydrone calibration/verification solution is created by adding 10g of quinhydrone to 1L of pH 4 or pH 7 buffer solution (ASTM D1498). The solutions are mixed on a magnetic mixing plate for a minimum of 15 minutes to create a saturated solution with undissolved crystals remaining. Quinhydrone solutions are usable for 8 hrs from the time of mixing.

- Light's solution consists of ferrous and ferric ammonium sulphate in sulphuric acid. The solution would rarely be used at SESD due to its high acidity and associated handling difficulty. Spent solutions with a pH<2 would be regulated as a hazardous waste. Light's is a highly poised solution that may allow a marginally functioning electrode to pass calibration.

- A prepared potassium iodide solution is available which has low toxicity and a long shelf life. The solution may stain clothing or surfaces if spilled.

3.2 Verification and Calibration

ORP instruments may be **verified** or **calibrated**, depending on the application. The approach chosen should be selected based on project needs and information presented in Section 2.4., Limitations. Standard laboratory practice in making ORP measurements is to **verify** the accuracy of the instrument prior to use, and this practice should be followed when true quantitative results

are required. In a **verification,** the instrument in its direct-reading mode is checked against a standard solution in a pass/no-pass test, and no corrections are applied to subsequent measurements. In most applications, the ORP information is used semi-quantitatively and for these applications, the instruments may be **calibrated** to the standard solutions. In an instrument **calibration**, the instrument probe is placed in the standard solution and the difference between the standard measurement and the known ORP value of the standard is used by the instrument to make adjustments to the subsequent measurements.

In **verification** of an ORP instrument, the instrument is set to absolute mV reading mode or the internal calibration offset is zeroed out. The instrument probe should then be placed in the standard solution and the reading verified to fall within +/-10mV of the predicted reading for the standard. Instruments with single-purpose electrodes are most suitable for this approach. If the instrument fails the verification, standard solution quality should be considered and instrument maintenance performed per the manufacturer's procedures.

In most SESD field practice, the end data use is semi-quantitative. In this case, the instruments can be **calibrated** to standard solutions appropriate for the project using the manufacturer's recommended procedure. One minute after the calibration, the instrument should display a stable reading within +/-10mV of the predicted reading. An instrument failing this test should be recalibrated to determine if the problem is inadequate equilibration time. In the event of continued instrument failure, aging or contamination of the standard solution should be considered. Subsequently the electrode should be serviced according to the manufacturer's procedures. Common service procedures include cleaning the platinum electrode with mild abrasives or acids and refilling or replacing the reference electrode.

Prior to a mobilization, all ORP instruments will be checked for proper operation and verified or calibrated against standard solutions. During the field mobilization, each instrument will be calibrated or verified prior to, and verified after, each day's use or deployment.

Even though it is not necessary to re-calibrate ORP instrument at regular intervals during the day, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an extreme temperature change or submersion of a filling solution port have impacted the instrument's performance. If an operational check is warranted, the field operator should follow the appropriate verification/calibration steps as described above.

The predicted ORP values of standard solutions will be obtained from the manufacturer of prepared solutions, literature, or appropriate values listed in this procedure. Care is in order, as the predicted ORP value is specific for the type of reference electrode used by the probe (either Ag/AgCl or calomel) and the molarity of the filling solution in the reference electrode. To use the solution with another electrode or filling solution, the expected ORP readings for the solution should be converted to Eh for the probes intended for the solution as per the Reporting section of this procedure. Then a table can be compiled for the electrode in use by subtracting the $E_{h,ref}$ for the electrode and filling solution in use. This will be done at the Field Equipment Center (FEC) for the solutions stocked.

Verification solutions should be managed per the manufacturer's directions regarding storage and handling. After instrument verification or calibration, the solution cannot be returned to the stock solution container, although a separate container of working solution can be maintained.

Spent solutions and working solutions should be returned from the field to the SESD laboratory for proper disposal by the SHEMP, or handled as directed by the SHEMP. Properly handled stock solutions may be returned to the FEC for use at that facility.

3.3 Measurement

ORP measurements should be conducted in a fashion that prevents the addition or loss of any potential oxidants or reductants. Results could be compromised by exposing the sample to air or allowing H_2S to off-gas from anoxic samples. Like dissolved oxygen measurements, ORP measurements should be conducted in situ or by using a flow-through cell evacuated of air (see the SESD Operating Procedure for Field Measurement of Dissolved Oxygen (SESDPROC-106, most recent version).

When using multi-parameter probes for ORP measurements, the general guidelines for probe deployment described in the SESD Operating Procedure for Field Measurement of Dissolved Oxygen (SESDPROC-106, most recent version) and the SESD Operating Procedure for In situ Water Quality Monitoring (SESDPROC-111, most recent version) apply.

ORP probes must be operated and maintained in accordance with the manufacturer's instructions. Reference electrodes in multi-parameter probes may require regular filling or replacement. Single parameter ORP electrodes may require regular filling and operation in an upright position to assure that proper salt bridge flow is maintained. Platinum electrode surfaces are easily contaminated and polishing or cleaning of the electrodes should be performed as recommended by the manufacturer.

Measurements in field logbooks should be recorded to the nearest mV. The type of reference electrode in use and its filling solution should be recorded in at least one logbook as part of the field project records.

ORP is a temperature sensitive measurement, but ORP instruments are not temperature compensated. Consequently, the media temperature should always be recorded at the same time as the ORP is recorded. Likewise, as ORP is often pH dependent, pH should also be recorded at the time of ORP measurement.

3.4 Reporting

In the absence of a specified reference scale, ORP data has no meaning. Therefore, the reference scale used should always be specified in reporting or discussing the ORP data. ORP measurements converted to a hydrogen scale can be reported as "E_h". Data reported as the direct field measurement without correction might be described as "ORP referenced to Ag/AgCl electrode" or "$E_{Ag/AgCl}$". The expectations of the data user should be ascertained or the measurements should be reported in both systems.

To apply corrections to obtain E_h from the direct field measurement, the known half-cell potential of the reference electrode is added to the recorded field ORP value:

$$E_{h,sample} = ORP_{sample} + \text{half-cell potential of reference electrode}$$

The following table, reproduced from Section 2.2, presents the half-cell potential of a silver/silver chloride reference electrode at various temperatures and with various molarities of KCl filling solutions.

Table 1

Half-cell Potential of Ag/AgCl reference electrode

derived from USGS NFM, Table 6.5.2 (9/2005)

	Molarity of KCl filling solution			
T(°C)	3M	3.3M*	3.5M	Sat/4M
10	220	217	215	214
15	216	214	212	209
20	213	210	208	204
25	209	207	205	199
30	205	203	201	194
35	202	199	197	189
40	198	195	193	184

*interpolated value

Note: YSI sondes and Thermo electrodes typically use 4M KCl filling solutions. Eureka sondes typically use 3.3M KCl filling solutions

Example:

A multi-parameter probe with a silver/silver chloride reference electrode and 4M KCl filling solution is used to record a stream ORP measurement of 146mV. The stream temperature is recorded as 15°C.

From the above table, the half-cell potential of an Ag/AgCl reference electrode filled with 4M KCl is 209mV at 15°C. Then:

$$E_{h,sample} = ORP_{Ag/AgCl,sample} + \text{half-cell potential of Ag/AgCl reference electrode}$$

$$E_{h,sample} = 146mV + 209mV$$

$$E_{h,sample} = 355mV$$

As noted in Section 3.3, Measurement, ORP measurements are sensitive to temperature, and may be sensitive to pH. As the instruments do not compensate for these parameters, ORP data should always be reported with the temperature and pH of the media at the time of measurement.

Final reporting values of Eh or ORP should be rounded to the nearest 10mV. The following spreadsheet formula can perform the rounding of an interim result located in spreadsheet cell 'A1':

=INT(A1/10+0.5)*10

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field pH Measurement**

Effective Date: January 29, 2013	**Number:** SESDPROC-100-R3

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-100-R3, *Field pH Measurement,* replaces SESDPROC-100-R2 **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Author was changed from Ron Phelps to Timothy Simpson. The EIB Branch Chief was changed from Antonio Quinones to Danny France. The EAB Branch Chief was changed from Bill Cosgrove to John Deatrick. The Field Quality Manager was changed from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3, last sentence:** Added "and for maintaining records of review conducted prior to its issuance." **Section 1.4:** Added reference to the SHEMP Manual. **Section 1.5.1:** Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used. **Section 2:** In the first paragraph, replaced "and" in the second sentence with "or". **Section 3.2:** Replaced the first paragraph with the following language for clarification purposes: "Many brands of instruments are commercially available for the measurement of pH incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. At a minimum, a two-point calibration should be conducted to ensure the accuracy of the meter. The following are basic guidelines for calibration/verification and are provided as an example: In Item #4, the first sentence, replaced "original buffer" to "appropriate buffer".	January 29, 2013

Item #5 was replaced with the following: "Once the meter has been properly calibrated and verified (steps 1-4 above), it is ready for use. Rinse the probe with de-ionized water and store it according to manufacturer's recommendations. Certain instruments may require being left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against a known calibration buffers if used for extended periods (> 4 hrs). **Section 3.4:** Revised the language to clarify procedures associated with operational checks. Item # 2 was converted to paragraphs 2.	
SESDPROC-100-R2, *Field pH Measurement,* replaces SESDPROC-100-R1 **Cover Page:** Author was changed from Marty Allen to Ron Phelps. **Revision History** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3** Changed Field Quality Manager to Document Control Coordinator. **Section 2** Added requirements for unattended deployment of in-situ monitoring equipment. **Section 3.1 and 3.2, 4** Clarified requirements for routine and NPDES compliance monitoring. **Section 3.2, 2 and 5** Added first sentence for clarification.	June 13, 2008

SESDPROC-100-R1, *Field pH Measurement*, replaces SESDPROC-100-R0 **General** Deleted all references to SOSA. Updated referenced procedures due to changes in title names and/or to reflect most recent version. Replaced "shall" with "will". **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 2** Added last paragraph regarding stopping measurements due to environmental conditions. **Section 3.4** Re-phrased procedure #2 for clarity.	November 1, 2007
SESDPROC-100-R0, Field pH Measurement, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field pH measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring the pH of aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain pH measurements of the media being sampled, and that another method must be used to obtain said measurements, the variant instrument and/or measurement procedure will be documented in the field logbook and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on SESD's local area network (LAN). The Document Control Coordinator is responsible for ensuring that the most recent version of the procedure is placed on SESD's LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

USEPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field pH measurements. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field pH measurements pertinent to the sampling event should be recorded in the field logbook for the event. All records, including a unique, traceable identifier for the instrument, such as a property number or serial number, should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010) and the SESD Operating Procedure for Equipment Inventory and Management, (SESDPROC-108).

Care should be taken not to contaminate standards and samples and verify the expiration date of all standards prior to use. All meters should be calibrated, operated and maintained according to the manufacturer's specifications.

2 Quality Control

All pH meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter is taken to the field, it will be properly calibrated or verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for *in-situ* measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in-situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field pH Measurement Procedures

3.1 General

pH is defined as the negative logarithm of the effective hydrogen-ion concentration. For routine work, a pH meter accurate and reproducible to within 0.2 Standard Unit (S.U.) is suitable. For NPDES compliance monitoring, the pH meter should be accurate and reproducible to within 0.1 S.U. Both meters should have a range of 0 to 14 S.U.s and be equipped with a temperature-compensation adjustment.

3.2 Instrument Calibration

Many brands of instruments are commercially available for the measurement of pH incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. At a minimum, a two-point calibration should be conducted to ensure the accuracy of the meter. The following are basic guidelines for calibration/verification and are provided as an example:

1. Verify the meter's internal temperature sensor (thermistor) against a National Institute of Standards and Technology (NIST) traceable thermometer and note any differences between the thermistor and the NIST-traceable thermometer in the logbook. If the temperatures do not agree within ± 4°C, the unit or probe must be repaired or replaced. Alternatively, if the meter can be used in a manual temperature compensation mode, the NIST-traceable thermometer may be used for temperature readings and the necessary corrections applied. Check and record the temperatures of the standards and the samples.

2. If the pH range of the sample is not known, the pH of the sample to be tested should be estimated either from historical data or by using a four-color pH indicator paper or equivalent. Using this information, calibrate the pH meter with the buffers that bracket the expected pH range. Buffer solutions are commonly pH 4, 7 and 10. It may be possible to configure the pH meter so that it can be standardized with buffers other than those in the default configuration.

3. Immerse the probe in the first buffer solution and calibrate the meter to read the correct pH. After the initial buffer calibration, calibrate the meter using other buffer solutions, as appropriate. Rinse the probe with de-ionized water and blot dry or otherwise remove excess rinse water between the different buffer solutions. Record the buffer values and temperatures used to calibrate the meter.

4. Rinse the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate buffer and read as a sample. If the meter reads within ± 0.2 S.U. of the known value of the buffer (for general applications such as ecological studies) or ± 0.1 S.U. (for regulatory applications such as NPDES or drinking Water programs), record the value indicated by the meter. If the meter is outside of the acceptable accuracy range, it should be recalibrated. If it is still outside of the acceptable accuracy range after the second calibration, the electrode and/or meter should be replaced.

5. Once the meter has been properly calibrated and verified (steps 1-4 above), it is ready for use. Rinse the probe with de-ionized water and store it according to manufacturer's recommendations. Certain instruments may require being left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against a known calibration buffers if used for extended periods (> 4 hrs).

6. Unless the manufacturer indicates that the meter maintains its calibration after being turned off, meters must be re-calibrated if they are turned off during their period of use.

3.3 Sample Measurement Procedures

These procedures should be followed when conducting field pH measurements of grab samples:

1. Collect a sample. If the meter's thermistor is to be used for the temperature of record for the measurement activity, the temperature should be read as soon as the reading stabilizes and prior to measuring the pH.

   Note 1: When the pH meter response is slow, unstable, or non-reproducible, it may be necessary to check the conductivity. If the conductivity is lower than 20 to 30 µmhos/cm, it is permissible to add 1 ml of 1M potassium chloride solution per 100 ml of sample to improve response time for the probe. Recheck the pH and record.

   Note 2: If the pH measurements are to be used for RCRA regulatory purposes and when the pH approaches the alkaline end (pH ≥ 11.0) of the scale, the pH measurements should be made by a qualified analyst using laboratory quality equipment to control the sample at 25°C ± 1°C.

2. Immerse the probe in the sample keeping it away from the sides and

bottom of the sample container. Allow ample time for the probe to equilibrate with the sample.

3. While suspending the probe away from the sides and bottom of the sample container, record the pH.

4. Rinse the probe with de-ionized water and store it in the manufacturer's recommended storage solution until the next sample is ready.

These procedures should be followed when conducting in-situ field pH measurements:

1. Place the probe into the media to be measured and allow the pH and temperature readings to stabilize. Once the readings have stabilized, record the measurements in the logbook.

2. When deploying meters for extended periods of time, insure the measurement location is representative of average media conditions.

3.4 Operational Check

Even though it is not necessary to re-calibrate pH meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.

1. While in use, periodically check the pH by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate buffer solution. If the measured pH differs by ≥ 0.2 S.U. or 0.1 S.U. (depending on the application) from the buffer solution, the meter must be re-calibrated.

A post-operation instrument verification check will be performed using the appropriate buffer(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Sampling and Measurement Procedures and Procedure Validation**

Effective Date: August 30, 2012	**Number:** SESDPROC-012-R3

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SESDPROC-012-R3, *Field Sampling and Measurement Procedures and Procedure Validation*, replaces SESDPROC-012-R2 **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the Author from Don Hunter to Bobby Lewis. Changed the Enforcement and Investigations Branch Chief from Archie Lee to Danny France. Changed the Ecological Assessment Branch Chief form Bill Cosgrove to John Deatrick. Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis. **Added Section 4** which describes when Competency testing should be conducted for implementing a new procedure after it has been validated and accepted.	August 30, 2012
SESDPROC-012-R2, *Field Sampling and Measurement Procedures and Procedure Validation*, replaces SESDPROC-012-R1 **General:** Corrected typographical errors. **Title Page:** Changed Enforcement and Investigations Branch Chief from Antonio Quinones to Archie Lee. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3:** Omitted reference to the H: drive. Changed the Field Quality Manager to the Document Control Coordinator.	December 18, 2009
SESDPROC-012-R1, *Field Sampling and Measurement Procedures and Procedure Validation*, replaces SESDPROC-012-R0 **Revision History:** Changed title for Antonio Quinones from Environmental Investigation Branch to Enforcement and Investigations Branch.	November 1, 2007
SESDPROC-012-R0, Procedure Development, Revision and Validation, Original Issue	October 10, 2007

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1 General Information

1.1 Purpose

SESD field branches operating procedures are reviewed periodically, and where necessary, revised to ensure continuing suitability and conformance with applicable requirements and industry standards. Additionally, there may be the occasion where a new sampling or measurement procedure is developed internally or adapted from an existing standard or non-standard procedure. This document defines the different classes of procedures and describes the process for:

- The development and validation of new SESD procedures; and
- The development, review/validation and approval process to be followed when adopting or revising standard or non-standard procedures.

All SESD sampling and measurement procedures in use as of February 5, 2007 are considered standard procedures and do not require validation.

1.2 Scope/Application

This document applies to all standard, non-standard and SESD-developed sampling and measurement procedures.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

The following definitions apply to this operating procedure.

1.4.1 Standard Procedure

This term applies to an established environmental sampling or measurement procedure used and accepted within the environmental industry.

1.4.2 Non-Standard Procedure

A non-standard procedure is an environmental sampling or measurement procedure that is not widely used or accepted within the environmental industry.

1.4.3 SESD-Developed Procedure

A new sampling or measurement procedure developed by the SESD field branches for internal use.

1.5 References

SESD Operating Procedure for Document Control (SESDPROC-001, most recent version)

2 Field Sampling and Measurement Procedures

2.1 Standard Procedures

SESD field investigators use standard procedures in the course of conducting field investigations. SESD procedures are subject to periodic review and where necessary, may be revised to ensure continuing suitability and conformance with applicable industry standards and requirements. SESD procedures undergoing periodic review that are updated with minor modifications are not subject to the validation process described in Section 3 of this procedure.

New standard procedures may be adopted for use by SESD field investigators. In addition, as state-of-the-art changes and new instruments, equipment, and procedures are developed and become available, it may be appropriate for SESD to develop and publish new operating procedures for field measurement and sampling activities.

New standard procedures adopted for use by the field branches will not be subject to an extensive validation as described in Section 3 of this procedure. New standard procedures will be evaluated to the extent necessary to ensure the result provided is equivalent to a currently used procedure or consistent results are achieved. Documentation supporting the use and acceptance of standard procedures adopted after February 5, 2007 will be maintained by the Field Quality Manager.

2.2 Non-Standard Procedures

When it is necessary to use a non-standard sampling or measurement procedure, the project leader will:

1. Inform the customer in writing (email or memo) that a non-standard procedure will be used;
2. Document that the customer is in agreement with the use of a non-standard procedure; and
3. Document in the project file the customer's request and the need for using a non-standard procedure (i.e., description of the project request and why it required the use of a non-standard procedure).

Non-standard procedures will be fully validated according to Section 3 of this procedure. If the non-standard procedure is not adopted into the scope of the field branches quality system prior to use, any results generated by the procedure must be clearly identified as being outside the scope of the quality system in the field investigation report.

If the field branches choose to include the non-standard procedure in the scope of their quality system, this will be done in accordance with the SESD Operating Procedure for Document Control (SESDPROC-001, most recent version).

2.3 SESD-Developed Procedures

When it is necessary for the field branches to develop procedures for their own use, it will be a planned activity and management will designate qualified personnel to develop the procedure. Management will also ensure adequate resources (time, equipment, funding, personnel, etc.) are available to aid in the development process. If a team of staff members is tasked with the procedure development, management will designate an individual to serve as the team leader. This individual will be responsible for facilitating the development process. The development process will consist of:

1. Review of relevant literature (manufacturers' documentation, scientific publications, etc.);
2. Development of an operating procedure; and
3. Validation of the procedure.

3 Validation

3.1 General

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. SESD will validate 1) non-standard procedures; 2) SESD developed procedures; 3) standard procedures used outside their intended scope; and 4) amplifications and modifications of standard procedures to confirm that the procedures are fit for the intended use. Validation will be as extensive as is necessary to meet the needs of the given application.

3.2 Validation Team

SESD field branches management will designate members of the validation team and ensure adequate resources are available to complete the validation. Members may consist of subject matter experts (SMEs) and representatives from both field branches, as appropriate. Management will designate one team member as the team leader. The team leader will serve as the point of contact for the team and facilitate the validation process. The team will be responsible for developing a validation plan that should address, at a minimum, the objective of the validation, acceptance criteria, the level of testing necessary and peer review. The team will update the validation plan, as necessary, during the validation process.

3.3 Field Testing or Evaluation

The validation team will develop an appropriate field test or evaluation for the operating procedure. A member or members of the validation team will be present during the field test or evaluation to record information on the conditions, performance, and practicality of the proposed procedure.

3.4 Assessment

After completion of the field test or evaluation, the validation team will assess the results using the acceptance criteria established by the team in the validation plan.

3.5 Acceptance/Rejection of Operating Procedure

If the assessment indicates that the operating procedure meets the acceptance criteria, the validation team will finalize the operating procedure as written, or with modifications. If the acceptance criteria are not met, the team may continue the validation by returning to earlier steps in the validation process, as appropriate.

3.6 Records

The validation team will document all decisions and actions. The following records will be generated during the validation process:

1. Validation plan
2. Relevant literature
3. Draft and final operating procedures
4. Results of field tests/evaluations
5. Results of the assessment

The validation team leader will be responsible for maintaining the records during the validation process. Upon completion of the validation, the records will be maintained by the Field Quality Manager.

4 Procedure Implementation

After a standard or nonstandard procedure has been assessed and accepted for use, the subject matter expert will provide training, to the appropriate staff, on how to correctly implement the operating procedure. Competency will be documented, in accordance with SESD's Competency and Proficiency Testing Operating Procedure (SESDPROC-006, most recent version) for each staff member who will be assigned to implement the operating procedure.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Sampling Quality Control**

Effective Date: February 5, 2013	**Number:** SESDPROC-011-R4

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SESDPROC-011-R4, *Field Sampling Quality Control*, replaces SESDPROC-011-R3 **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Archie Lee to Danny France. The Ecological Assessment Branch Chief was changed from Bill Cosgrove to John Deatrick. The FQM was changed from Liza Montalvo to Bobby Lewis. **Section 2.5:** Modified the statement to read: Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the BFEM. **Section 3.3.3**:Modified section to read that SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. Added Note for bullet 1. Modified bullet number 9 to read Water Filter Blank. **Section 4.1:** Modified first sentence to read - Records will be kept by FEC personnel. Modified bullet 1 to only apply to the FEC water system. Section 4.1, #5: - Added tubing to bullet number 5. Created bullet number 6. Moved last two sentences from bullet number 5 to create bullet number 7. **Section 4.2:** Moved testing requirements for Teflon® tubing from bullet number 1 to bullet number 6. Added requirements to bullets 2 and 4 that organic-free water for VOC samples is provided by the ASB laboratory. **Section 4.4.1:** Changed requirement for the EAB QA Officer from submitting an annual QA report to a quarterly report. Modified paragraph to read - Each branch QAO will compile a quarterly report of field quality assurance data and forward the report to the FQM.	February 5, 2013
SESDPROC-011-R3, *Field Sampling Quality Control*, replaces SESDPROC-011-R2 **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo.	October 15, 2010

Section 1.2: Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted reference to the H: drive of the LAN. **Section 2.9**: On the first paragraph, replaced the word "will" with "should" on the two instances where "will" was mentioned. **Section 4.2, Subsection 1**: Removed volatiles from list of analyses for 1-liter amber containers. Added volatile organic compound to list of analyses for 8-oz. clear glass containers.	
SESDPROC-011-R2, *Field Sampling Quality Control*, replaces SESDPROC-011-R1 **Revision History** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3** Changed Field Quality Manager to Document Control Coordinator. **Section 2.6** Added item #8. **Section 4.1** Added clarifying language to item #5. **Section 4.2** Added "volatiles" to item #1. Added "Note" at end of item #1. **Section 4.4.1** Changed "quarter" to "year" in second paragraph.	January 28, 2008
SESDPROC-011-R1, *Field Sampling Quality Control*, replaces SESDPROC-011-R0. **Section 1.3** Modified requirements for maintaining official copy of procedure. **Section 1.4** Stated that variability should be accounted for depending on the data quality objectives for the study. Added definitions for branch field equipment manager and de-ionized water. Removed definitions for representative sample, sample representativeness, accuracy, precision, and bias. **Section 2.5** Records of sample preservation, including ice, should be documented in the field log books. **Section 2.6**	October 19, 2007

Added the SESD glove and bottle return policy. The glove and bottle return policy was previously in SESDPROC-108-R0, *Equipment and Supply Management*. **Section 3.3** Stated that variability samples will be collected in accordance to project DQOs. Removed definition for "major project". **Section 3.5** Added double volume requirement for SVOC/pesticide/PCB MS/MSD sample collection. **Section 4.1** Modified requirements for servicing both field and FEC organic-free water systems from quarterly to at least once per 180 days. **Section 4.2, Subsection 1** Added analytical requirements for testing of Teflon® tubing. **Section 4.2, Subsection 5** Modified Silastic® or Tygon® tubing testing requirements to state that new lot are required to be tested quarterly. **Section 4.4.2** Modified requirements that project leaders provide final report to QAO. Project Leaders will provide QA data to QAO.	
SESDPROC-011-R0, *Field Sampling Quality Control*, Original Issue	February 5, 2007

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Contents

1 General Information

1.1 Purpose

This document describes procedures established to ensure the quality of SESD field sampling activities, including Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations. Collectively, these procedures ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures, resulting in the collection of representative samples. Strict adherence to these procedures forms the basis for an acceptable field sampling quality assurance program.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling samples in the field and when preparing sampling equipment for SESD field investigations. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Sample

A part of a larger lot, usually a volume, area, period or population.

1.4.2 Variability

The range or "distribution" of results around the mean value obtained from samples within a population. There are three types of variability which should be measured or otherwise accounted for in field sampling, depending on the data quality objectives (DQO) for the study:

1. Temporal Variability

   Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

2. Spatial Variability

   Spatial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discreet "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

3. Sample Handling Variability

   Sample handling variability is the range of results due to the sample collection and handling techniques used by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

1.4.3 Grab Sample

An individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

1.4.4 Composite Sample

A sample collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or “aliquots”) which are combined or "composited." A composite sample represents the average characteristics of the population under consideration. Four types of composite samples are listed below:

1. Time Composite (TC) – a sample comprised of a varying number of discrete samples or “aliquots” collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.

2. Flow Proportional Composite (FPC) – A sample consisting of discrete samples or “aliquots” collected at a rate proportional to flow. The aliquots are collected during the compositing period by either a time-varying/constant volume (TV/CV) method (“automated flow proportioning”) or a time-constant/varying volume (TC/VV) method (“manual flow proportioning”). The TV/CV method is typically used with automatic

samplers that are paced by a flow meter. The TC/VV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is typically used when sampling wastewater.

3. Areal Composite – a sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

4. Vertical Composite – a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

1.4.5 De-ionized Water

Tap water that has been treated by passing it through a standard de-ionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. De-ionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for de-ionized water.

1.4.6 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager (BFEM) will be designated for the Enforcement and Investigations Branch (EIB) and the Ecological Assessment Branch (EAB).

1.5 References

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005-most recent version)

SESD Operating Procedure for Competency and Proficiency Testing, (SESDPROC-006, most recent version)

SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108-most recent version)

SESD Operating Procedure for Sediment Sampling (SESDPROC-200-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206-most recent version)

SESD Operating Procedure for Soil Sampling (SESDPROC-300-most recent version)

SESD Operating Procedure for Waste Sampling (SESDPROC-302-most recent version)

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version

Loan-In Form (SESDFORM-011, most recent version)

SESD Operating Procedure for Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209, most recent version)

SESD Operating Procedure for Training (SESDPROC-007-most recent version)

SESD Operating Procedure for Corrective Action (SESDPROC-009-most recent version)

SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101-most recent version)

2 Field Sampling Quality Control Considerations

This section provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all of the standard operating procedures outlined in this subsection forms the basis for an acceptable sampling quality assurance program.

2.1 Experience Requirements

There is no substitute for field experience. This field experience will be gained by on-the-job training using the "buddy" system. Each new investigator will accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators. Specific requirements covering experience, competency and proficiency are found in the SESD Operating Procedure for Competency and Proficiency Testing (SESDPROC-006) and SESD Operating Procedure for Training (SESDPROC-007).

2.2 Traceability Requirements

All sample collection and measurement activities will be traceable through field records to the person collecting the sample or making the measurement. All maintenance and calibration records for sampling and measurement equipment (where appropriate) will be kept so that they are similarly traceable. The SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108) contain specific procedures to be followed that ensure traceability.

2.3 Chain-of-Custody

Specific chain-of-custody procedures are included in SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005). These procedures will ensure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags or labels, field notes, and any other recorded information for accuracy. Additionally, the SESD FQM will randomly conduct reviews of project files to ensure that quality procedures are being followed.

2.4 Sampling Equipment Construction Material

Sampling equipment construction materials can affect sample analytical results. Field investigators will ensure the sample equipment construction material will not introduce contaminants to the sample being collected.

2.5 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM). Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the BFEM. All samples requiring preservation should be preserved immediately upon collection in the field. Records of sample preservation, including ice, will be documented in the field log books.

Samples that **should not** be preserved in the field are:

1. Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage or other source samples from hazardous waste sites are not to be preserved with any chemical.

2. Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved according to the ASBLOQAM.

All samples preserved with chemicals will be clearly identified by indication on the sample tag or label that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason. Samples shipped by air will not be preserved with nitric acid, hydrochloric acid, sodium hydroxide or sulfuric acid in excess of the amount specified in the ASBLOQAM.

2.6 Sample Collection Precautions

In order to prevent cross-contamination during sample collection, the following precautions will be taken:

1. A clean pair of new, non-powdered, disposable latex gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.

2. Sample containers for source samples or samples suspected of containing high concentrations of contaminants will be placed in separate plastic bags immediately after collecting, tagging, etc.

3. If possible, environmental (low concentration) samples and source or waste samples (high concentration) should be collected by different field teams. If different field teams cannot be used, all environmental samples should be collected first and placed in separate ice chests or shipping containers. Samples

of waste or highly contaminated samples should never be placed in the same ice chest as environmental samples. Ice chests or shipping containers for source or waste samples or any samples suspected to contain high concentrations of contaminants will be lined with new, clean, plastic bags.

4. If possible, one member of the field sampling team should record all of the field notes, collect GPS data, etc., while the other members collect the samples.

5. When sampling surface water and sediment at the same location, the water sample should always be collected before the sediment sample is collected.

6. Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.

7. Investigators should use equipment constructed of Teflon®, stainless steel, or glass that has been properly pre-cleaned according to either the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compounds analyses. Teflon® or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC should <u>not</u> be used to collect samples for trace organic compounds analyses.

8. Field investigators should ensure the sample containers they are using have been verified as suitable for the analyses that will be conducted on the samples through the quality control procedures discussed in Section 4 of this procedure.

Upon returning from the field, un-used sample containers will be examined by project leaders to determine whether bottles should be discarded, recycled or re-shelved for use on other projects. A load-in form (SESDFORM-011) will be completed and signed by project leaders to identify the future use of sample containers returning from the field. Opened boxes of sampling containers that can be re-used, will be segregated from sealed boxes of new containers.

Opened bags of latex gloves returning from the field will be segregated from unopened gloves and will not be re-used for sample collection on other projects.

2.7 Sample Handling and Mixing

Once a sample has been collected, it may have to be transferred into separate containers for different analyses. Sample transfer should be done as soon as possible. If necessary, aqueous samples may be collected into a single, larger container for homogenization and transferred into individual sample containers. However, aqueous samples collected for volatile organic compounds, oil and grease, bacteria, sulfides and phenols analyses may not be transferred using this procedure.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually.

2. Two quarters should then be mixed to form halves.

3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction and occasionally turning the material over.

2.8 Special Handling of Samples for Volatile Organic Compounds Analysis

Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon®-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the field investigation.

The vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Soil and sediment samples for VOC analyses should be collected and handled as specified in the SESD Operating Procedure for Soil Sampling (SESDPROC-300), Waste Sampling (SESDPROC-302) or the SESD Operating Procedure for Sediment Sampling (SESDPROC-200). Soil and sediment samples collected for VOC analyses should <u>not</u> be mixed.

2.9 Sample Storage and Transport

After collection, sample handling should be minimized. Field investigators should use extreme care to ensure that samples are not contaminated during storage. Environmental and waste samples are typically stored in coolers. To reduce the risk of cross contamination, smaller sample containers such as 8 ounce glass jars, 40 ml VOA vials, and one-liter amber bottles should be placed inside of sealed, plastic bags before being placed in the cooler. If ice is required for preservation of the samples, the ice should be contained in a plastic bag or some equivalent container to prevent the potential for cross contamination of the samples by water produced from melting ice. If ice is used, the coolers should be checked regularly and water should be drained as needed. Custody of samples will be maintained according to the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

Samples will either be transported to the analytical laboratory by field investigators or shipped by common carrier. Shipping of samples will be conducted in accordance with the SESD Operating Procedure for Packing, Marking, labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209).

3 Quality Control Samples

Quality control samples are collected during field studies for various purposes, among which are to isolate site effects (control samples), to define background conditions (background sample), and to evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples, etc.).

3.1 Control Sample

A control sample is typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

3.2 Background Sample

A background sample (usually a grab sample) is collected from an area, water body or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

3.3 Variability Samples

Variability may be defined as a variation in concentrations of compounds or analytes across a site or area of investigation or variations, across time, of waste streams or surface water bodies. Variation can also be introduced during sample handling. The following procedures are used to assess and evaluate variability. When appropriate, spatial duplicate grab and/or composite samples should be collected during investigations and studies in accordance to the project DQOs. In general, no more than ten percent of all samples should be collected as spatial duplicates.

3.3.1 Spatial Variability Duplicate

The following spatial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples. They should also be preserved in the same manner and submitted for the same analyses as the required samples.

Spatial variability duplicate samples are typically collected during investigations where samples are collected from grids that are positioned at fixed intervals over the study area and a sample collection pattern is established within the grids. Spatial variability duplicate samples are collected using the same compositing pattern as the original sample and are collected within the same general area of representativeness, however the pattern is shifted relative to the original aliquot locations. This amount and direction of shift for the duplicate sample is

dependent upon the size of the grid or area being sampled and should be specified in the QAPP for the investigation. Data from spatial duplicates will be examined by the investigation project leader to determine if the observed spatial variability is acceptable, based on the investigation or study objectives.

3.3.2 Temporal Variability Duplicate

When appropriate, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be similar to the time or span of time specified for the original sample in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

3.3.3 Sample Handling Variability

The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

Split Samples

Split samples will be collected by initially collecting twice as much volume as is normally collected. The material will be apportioned, after mixing, if appropriate, into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample," the other designated as a "split sample." Data from the split samples will be examined by the project leader to assess sample handling variability. On large studies (more than 20 samples collected), a minimum of 5 percent, but no more than 10 percent, of all samples will be collected as split samples unless required by site data quality objectives.

Blank Samples

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

1. Water Sample VOC Trip Blank - A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Sealed preserved (or unpreserved, if unpreserved vials were used during the investigation) 40-ml VOC vials will be transported to the field. Two sealed VOC vials will be submitted per trip blank sample. At least one trip blank sample will be submitted per sample shipment. Trip blanks will be prepared by lab personnel. Investigators should submit their request for trip blanks at least one week in advance of scheduled field investigations and inspections and never (except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled

inspection/investigation. These trip blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample labels and Chain-of-Custody Records as trip blanks.

2. Soil/Sediment Sample VOC Trip Blank - A soil/sediment sample VOC trip blank is required for every study where soil and/or sediment samples are collected for VOC analysis. The required containers are specified the USEPA Region 4 ASBLOQAM. The request and pick up of the soil blank sample will be the same as for the water trip blank. En Core® containers will be transported to the field. These blanks will be handled and treated by field investigators in the same manner as the soil samples collected for VOC analysis on that particular study. These samples will be clearly identified on sample labels and Chain-Of-Custody Records as trip blanks. Two sealed En Core® containers will be submitted per trip blank sample. At least one set of trip blank samples will be submitted per sample shipment.

The following blanks are prepared in the field:

1. Sample Preservative Blanks - SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. If preservative blanks are collected, sample containers will be filled with de-ionized water by SESD personnel and transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample labels and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. In addition, one preservative blank will be collected for each multi-use bottle of preservative used.

   Note: The deionized water will be generated from a water treatment unit provided by the SESD laboratory.

2. Equipment Rinsate Blanks - Equipment rinsate blanks will be collected whenever field decontamination of equipment to be re-used in sampling activities is performed.

   When field cleaning of equipment is required during a sampling investigation, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic-free water. The rinse water will be collected and submitted for analyses of all

constituents for which normal samples collected with that piece of equipment are being analyzed.

3. Organic-Free Water System Blanks - When using a portable organic-free water generating system in the field, a sample of the water generated by the system will be collected at least once during each week of operations. Based on the objectives of the study or investigation, it may be appropriate to collect a sample of the raw source water. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.

4. Material Blanks - When construction materials are being used on a site in such a way as to have a potential impact on constituent concentrations in the sample, a sample of each material will be submitted for analysis.

   Note: For drilling operations where materials are shipped directly to the site from the supplier, see SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101) for material blank collection and reporting requirements.

5. Automatic Sampler Blanks - In general, cleaning procedures outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) should be adequate to ensure sample integrity. However, it is the standard practice of the Environmental Investigations Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds and metals analyses. Automatic sampler blanks for other standard analyses may be submitted in the event of a special investigation (e.g., criminal or civil).

6. Field Blank - A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

7. Temperature Blank - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6EC (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory. No temperature blank is necessary for waste samples since waste samples do not require ice for preservation.

8. Wipe Sample Blank - A wipe sample blank is a sample of the material and solvent used for collecting wipe samples. The blank is handled, packaged and transported in the same manner as all other wipe samples with the exception that it is not exposed to actual contact with the sample medium.

9. Water Filter Blank - When filters are used for sampling a dissolved constituent, de-ionized water should be run through at least one filter from each lot and the filtered water submitted for the same analyses. When filters are used for chlorophyll sampling, the filter should be prepared using de-ionized water and submitted for the same chlorophyll analysis.

3.4 Spikes

Spike samples are used to measure bias due to sample handling or analytical procedures. Spike samples are typically used by SESD to evaluate the performance of contract laboratories and are shipped directly to the CLP laboratory by the ESAT contractor.

3.5 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses

Matrix spike and matrix spike duplicate (MS/MSD) samples will be submitted to the laboratory for volatile organic compounds, extractable organic compounds, pesticides/PCBs and/or herbicides analyses from at least one sampling location per project and laboratory used. One MS/MSD sample should be collected per 20 samples per media collected.

Additional volume will be required for the soil MS/MSD samples. Semi-volatile organic compounds, pesticides, and PCB analyses of soil/sediment samples require the collection of one additional eight ounce glass jar. For VOC soil/sediment samples, triple volume, i.e., nine En Cores® or nine 40 ml vials with syringe collected sample, is needed for the MS/MSD samples.

Additional volume will be required for the water MS/MSD samples. For routine full scan analysis, i.e., extractable organic compounds, pesticides and PCBs, four one-liter amber containers provide the required sample volume. Eight containers, therefore, should be submitted for the MS/MSD sample. For VOC water samples, a total of six 40-ml vials should be collected.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" or “MS/MSD” on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.6 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses

A matrix spike sample and a duplicate sample (MS/MSD) will be submitted to the laboratory for inorganic analyses from at least one sampling location per project and laboratory used. One matrix spike and duplicate sample should be collected per 20 samples per media collected per laboratory.

Soil/sediment and water samples collected for inorganic analyses will normally have sufficient sample volume to perform the matrix spike analyses without requiring the collection of extra sample volume. The project leader should designate a sample, typically one considered to be representative of background or relatively uncontaminated conditions, as the matrix spike sample. For water samples, the sample volume collected will normally provide adequate volume for the MS/MSD analyses.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. MS/MSD samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.7 Special Quality Control Procedures for EPA Contract Laboratories

On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by either the Region 4 SESD laboratory or contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established procedures. The contract laboratory involved <u>will not be notified</u> that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 SESD laboratory.

3.8 Special Quality Control Procedures for Dioxins and Furans

The Region 4 laboratory does <u>not</u> conduct in-house analyses for dioxins and furans. Dioxin and furans analyses are conducted by contract laboratories. The Region 4 laboratory may accept environmental samples (soil, sediment, groundwater and surface water) suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), as long as suspected PCDD and PCDF contamination is not due to RCRA hazardous waste classified as F020-023 and/or F026-028. If these environmental samples are not contaminated with an F020-023 and/or F026-028 waste, it may be analyzed for parameters other than dioxin and furans. Environmental samples known or suspected to be contaminated with the RCRA hazardous waste F020-023 and or F026-028 will not be accepted.

NOTE: Environmental samples suspected of being contaminated with RCRA hazardous waste classified in 40 CFR, 261.31 as F032 will be accepted. The F032 waste is defined as wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations. The F032 listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and or pentachlorophenol. Prior to a sampling event, the project leaders should consult with the Analytical Support Branch Sample Control Coordinator to determine if the Region 4 laboratory can accept the samples. The Region 4 SESD laboratory should also be consulted for the current quality control procedures for dioxins and furans samples prior to a sampling investigation.

4 Internal Quality Control Procedures

The focus of this section is on Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations, as well as, field data generated under the specific sample collection quality control procedures discussed in Section 2. Quality control checks of these operations ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures.

4.1 Traceability Requirements

Records, will be kept by FEC personnel documenting the dates of operations and the person performing operations for the following:

1. Organic-Free Water System Maintenance (FEC System) - Maintenance on the FEC organic-free water system will be performed at least once per 180 days.

2. Air Monitoring Safety Instrumentation Checkouts - Pre-loadout checks on safety monitoring instrumentation will be recorded each time they are performed. Discrepancies will be immediately reported to the Branch Safety Officer.

3. Self Contained Breathing Apparatus (SCBA) Checkouts - Pre-loadout checks on SCBAs will be recorded when they are performed. SCBA checkouts will be performed at least once per calendar quarter in the absence of loadout requests. Any discrepancies will be reported immediately to the Branch Safety Officer.

4. Other Equipment Maintenance - Maintenance performed on equipment other than that listed above will be accordance to the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). All required repairs will be reported to appropriate Branch Field Equipment Manager.

5. Tubing, Sampling Containers and Latex Gloves - The Enforcement and Investigations Branch Quality Assurance Officer (EIB QAO) is responsible for conducting verification sampling for tubing, sample containers, and latex gloves that are used during field investigations. Upon receipt, the tubing, containers and gloves are placed in the quarantine room at the FEC. A record is kept of the lot numbers for each shipment received. The EIB QAO will collect blank samples from tubing, containers and gloves within each lot received and will review the results to ensure the sample containers and gloves are suitable for use during field investigations. Once the supplies are deemed suitable, the EIB QAO will release the items for use.

6. Chemical preservatives commercially purchased will be tested prior to use. Each lot of chemical preservative will be tested for the appropriate analytes by either the BFEM or Branch QAO. Once released by Branch QAO, the preservatives can be used in the field.

7. Equipment - All equipment cleaned and wrapped for field use will be marked with the date on which preparation was completed. Equipment will be stored at the FEC in specified areas to minimize the risk of contamination while awaiting use.

4.2 Specific Quality Control Checks

When collecting samples during field investigations, it is necessary to take measures to prevent cross contamination to ensure the integrity of the data generated. The field branches conduct verification sampling of sample containers, gloves, sampling equipment, tubing and water utilized during field investigations as one of these measures. At least once per calendar quarter, the EIB QAO will conduct the following checks and issue a written report to the EIB Branch Chief and Field Quality Manager with the results.

1. Collect and submit for analyses samples of each new lot of containers received, Bottles from each lot will be tagged and sealed, then submitted for the following analyses:

    1. 1-liter Amber – extractable organics, pesticides, and PCBs.

    2. 8-oz. Clear Glass – metals, cyanide, extractable organics, pesticides, PCBs, and volatile organic compounds.

    3. 1-Liter Polyethylene – metals and cyanide.

    NOTE: In addition to the quality control checks listed above, samples may be collected during field investigations for classical inorganic parameters such as nitrates, nitrites, sulfides, etc. Due to the detection levels generally required for these parameters, it is unlikely that cross contamination may occur in association with the sample containers and sampling equipment used during sample collection. Therefore, classical inorganic analyses are not conducted as part of the routine quality control checks. If the data quality objectives require additional quality control checks, bottles will be submitted to the laboratory for analyses.

2. Collect and submit for analyses a rinsate blank for each new lot of latex gloves received during the calendar quarter. Samples will be collected as rinse blanks using organic-free water. The rinsate will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. A new glove will be rinsed for each parameter (e.g., one glove for the VOC sample, another glove for metals, etc.) to avoid dilution of potential contaminants on the gloves. Water for the VOC samples should be provided by the ASB laboratory.

3. Collect and submit for analyses a sample of water from the FEC organic-free water system. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs.

4. Collect and submit for analyses a rinsate blank of at least one piece of sampling or sample related equipment stored at the FEC. The sample will submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

5. Collect and submit for analyses a rinsate blank for each new lot of Silastic® or Tygon® tubing used in peristaltic pump head. The sample will be submitted for metals and cyanide analysis.

6. Teflon® tubing – Collect and submit for analyses a rinsate blank for each new lot of Teflon® tubing received. Rinse blanks will be collected through the Teflon tubing The sample will be submitted for metals, cyanide, extractable organics, volatile organic compounds, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

4.3 Quality Control for Special Order Equipment and Supplies

Some equipment and supplies ordered for specific projects are received in what can be considered ready to use condition. In order to ensure the integrity of these materials, an equipment rinsate blank will be collected from at least one item in each lot. The equipment and supplies will not be used until the QAO has reviewed the analytical data for the blanks and released the items.

4.4 Quality Control Evaluation and Corrective Action

All field investigation reports will contain a clearly identified section where the results for all field generated quality control (QC) samples are discussed and reported. Quality control data review includes but is not limited to detections of organic and inorganic compounds at any concentration in quality control blanks (i.e., trip blanks, equipment rinsate blanks, portable organic-free water system blanks, etc.).

All detections of organic and inorganic compounds will be immediately reported to the appropriate branch QAO. The project leader will analyze of the results to determine if the source of contamination can be identified. If the source of contamination cannot be determined by the project leader, the branch QAO will conduct an additional review of the results to assess the source of contamination. If the source of contamination cannot be determined, the branch QAO will monitor all quality control results generated by the branch and assess the data for trends of contamination.

If it is determined by the project leader and the branch QAO that the contamination adversely impacts the data collected during the investigation, the project leader will report the results to their Section Chief and the FQM. The project leader, in consultation

with management, will determine whether the impacted data are usable or should be rejected. If data are rejected, the project leader and their management will determine whether samples must be recollected.

Data reported to the FQM will be analyzed to determine if the contamination is due to non-conforming work. If it is determined by the FQM, in consultation with management, that the contamination is due to non-conforming work, a corrective action is warranted and will be selected and implemented in a timely manner. If a corrective action is required, it must be implemented and reported according to the SESD Operating Procedure for Corrective Action (SESDPROC-009). If contamination is not due to non-conforming field work, then the source of contamination will be identified, if possible, and documented by the FQM. If the source of contamination cannot be determined, FQM will monitor all quality control results generated by SESD and assess the data for trends of contamination.

4.4.1 Quality Assurance Reports

It is each project leader's responsibility to ensure that a copy of the quality assurance data from each field investigation report is provided to their respective branch QAO. Each branch QAO will compile a quarterly report of field quality assurance data and forward the report to the FQM.

The FQM will prepare an annual quality assurance report based on the reports provided by the branch QAOs. This report will be distributed to all field investigators each year and will document and discuss all quality control issues or trends identified during the data review. This report will be retained by the FQM to document that QC measures have been taken, that the QC measures are appropriate, that the QC results are acceptable or, if not, that corrective actions were taken.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Specific Conductance Measurement**

Effective Date: August 30, 2012	**Number:** SESDPROC-101-R5

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-101-R5, *Field Specific Conductance Measurement*, replaces SESDPROC-101-R4 **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. **Section 3.4:** Added paragraphs 1 and 2 concerning performing operational checks during the day.	August 30, 2012
SESDPROC-101-R4, *Field Specific Conductance Measurement*, replaces SESDPROC-101-R3 **Cover Page:** Hunter Johnson was added as co-author. The EIB Branch Chief was changed from Archie Lee to Danny France. The FQM was changed from Liza Montalvo to Bobby Lewis. **Section 3.2:** Replaced the first paragraph with the following language for clarification purposes: "Many brands of instruments are commercially available for the measurement of specific conductance incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate." In the second paragraph, revised the language of the first sentence to clarify documentation procedures associated with the effect of temperature on conductivity. Replaced the second sentence with "The following are basic guidelines for calibration/verification and are provided as an example:" The note found in Item # 3 was revised to clarify the relationship between calibration standards and the anticipated specific conductance. In Item # 6, the third sentence was replaced with the following: "Certain meters may require that the instrument be left on until all sample measurements are performed and the results are recorded." In the forth sentence, replaced "it is recommended" with "certain instrument manufacturers recommend." **Section 3.3**: In the first sentence, replaced "must" with "should."	January 13, 2012

SESDPROC-101-R3, *Field Specific Conductance Measurement*, replaces SESDPROC-101-R2 **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Author was changed from Ron Phelps to Timothy Simpson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Section 3.1:** Added "microsiemens per centimeter (µS/cm)" in addition to micromhos per centimeter (µmhos/cm) to the first sentence. **Section 3.2:** Item #3 - Added the following statement: "Fresh standards should be used for each calibration. Item #4 - Added language related to the auto-recognition of standards during meter calibration (first 3 sentences). Added items #5 and #6.	August 12, 2011
SESDPROC-101-R2, *Field Specific Conductance Measurement*, replaces SESDPROC-101-R1 **Cover Page:** Author was changed from Marty Allen to Ron Phelps. **Revision History** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3** Changed Field Quality Manager to Document Control Coordinator. **Section 2** Added requirements for unattended deployment of in-situ monitoring equipment. **Section 3.2** Added sentence to paragraph 2 to identify minimum requirements for calibration/verification. **Section 3.3** Moved operational check to Section 3.4.	June 13, 2008

SESDPROC-101-R1, *Field Specific Conductance Measurement*, replaces SESDPROC-101-R0 **General** Deleted all references to SOSA. Updated referenced procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 2** Added last paragraph regarding stopping measurements due to environmental conditions.	November 1, 2007
SESDPROC-101-R0, *Field Specific Conductance Measurement*, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes procedures, methods and considerations to be used and observed when conducting field specific conductance measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when measuring the specific conductance of aqueous phase environmental media in the field. On the occasion that SESD field investigators determine that any of the procedures described in this section cannot be used to obtain specific conductance measurements of the media being sampled, and that another method must be used to obtain said measurements, the variant instrument and/or measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field specific conductance measurements. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field specific conductance measurements pertinent to the sampling event, including a unique, traceable identifier for the instrument, such as a property number or serial number, should be recorded in the field logbook for the event. All records should be entered according to the procedures outlined in the SESD Operating Procedure Logbooks (SESDPROC-010, most recent version).

Care should be taken to not contaminate standards and samples and verify the expiration date of all standards prior to use. All meters should be calibrated, operated and maintained according to the manufacturer's specifications.

2 Quality Control

All specific conductance meters will be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108, most recent version). Before a meter is taken to the field, it will be properly calibrated or verified, according to Section 3.2 of this procedure, to ensure it is operating properly. These calibration and verification checks will be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to ensure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in-situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Specific Conductance Measurement Procedures

3.1 General

Specific conductance is a measure of the ability of an aqueous solution to conduct an electric current and is customarily reported in microsiemens per centimeter (µS/cm) or micromhos per centimeter (µmhos/cm) at 25°C. It is important to note that if the specific conductance measurements are for NPDES reporting purposes, the meter and conductivity cell should be verified by comparing against a laboratory meter with a platinum-electrode type conductivity cell.

3.2 Instrument Calibration and Verification

Many brands of instruments are commercially available for the measurement of specific conductance incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate.

Conductivity is affected by temperature; therefore, for instruments that do not automatically compensate for temperature, the user should document temperature first so that appropriate adjustments can be made in accordance with the manufacturer's instructions and/or method. The following are basic guidelines for calibration/verification and are provided as an example:

1. Verify the meter's internal temperature sensor (thermistor) against a National Institute of Standards and Technology (NIST) traceable thermometer and note any differences between the thermistor and the NIST-traceable thermometer in the logbook. If the temperatures do not agree within ± 4°C, the unit must be repaired or replaced. Alternatively, if the meter can be used in a manual temperature compensation mode, the NIST-traceable thermometer may be used for temperature readings and the necessary corrections applied. Check and record the temperatures of the standards and the samples.

2. Rinse the probe with de-ionized water and blot dry before conducting the following calibration and verification checks.

3. Immerse the probe in the first standard solution and calibrate or verify the meter against that solution. Fresh standards should be used for each calibration. After the initial standard, calibrate and/or verify the meter using additional standards, as appropriate. Rinse the probe with de-ionized water and blot dry or otherwise remove excess rinse water between the different standards. Record the standard values/temperatures used to calibrate or verify the meter.

   Note: Some instruments require that calibration standards reflect the

anticipated specific conductance of the media being measured.

4. Some meters will auto-recognize standards during calibration. For example, the Orion Star Series meter will auto-recognize standards 1413 µS/cm, 100 µS/cm and 12.9 mS/cm. If the meter is calibrated in a manner where it does not auto-recognize the standard, and the meter is not accurate to within ± 10 % of the standard solution(s) known values, the meter or probe should be repaired or replaced. If this condition can be corrected by adjusting the cell constant of the probe, refer to the instruction manual and make the adjustment.

5. After calibration is complete, place the probe back into the calibration standard used and record a post-calibration reading. Record a post calibration reading for each standard used. If the meter is not accurate to within ± 10 % of the standard solution(s) known values, it should be recalibrated. If it is still outside of the acceptable accuracy range after the second calibration, the probe and/or meter should be replaced.

6. Once the meter has been properly calibrated and verified (steps 1-5 above), it is ready for use. Rinse the probe with de-ionized water and store it in the manufacturer's recommended storage solution. Certain meters may require that the instrument be left on until all sample measurements are performed and the results are recorded. When collecting measurements from grab samples, certain instrument manufacturers recommend that an intermediate check(s) be performed by periodically checking the meter against the known calibration standards if used for extended periods (> 4 hrs).

3.3 Sample Measurement Procedures

The following procedures should be followed when conducting field specific conductance measurements of grab samples:

1. Collect the sample, check and record its temperature.

2. Correct the instrument's temperature adjustment to the temperature of the sample (if required).

3. Immerse the probe in the sample keeping it away from the sides and bottom of the container. It is important that the center portion of the probe be wetted by the sample.

4. Allow meter to stabilize. Record the results in a logbook.

5. Rinse probe with de-ionized water.

The following procedures should be followed when conducting in-situ field specific conductivity measurements:

1. Place the probe into the media to be measured and allow the specific conductivity and temperature readings to stabilize. Once the readings have stabilized, record the measurements in the logbook.

2. When deploying meters for extended periods of time, ensure the measurement location is representative of average media conditions.

3.4 Operational Checks

Even though it is not necessary to re-calibrate conductivity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an extreme temperature change, have impacted the meter's performance. If an operational check is warranted, the following procedures should be followed to ensure that the performance of the meter has not changed.

Check the conductivity meter with fresh conductivity standard. Rinse the conductivity probe with deionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate conductivity standard. If the measured conductivity value is not with ± 10% of the standard, the probe should be re-calibrated. If the probe is still not within ± 10% of the standard, the probe should be repaired or replaced. These measurements must be recorded in the field logbook.

A post-operation instrument verification check should be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Temperature Measurement**

Effective Date: February 4, 2011	**Number:** SESDPROC-102-R3

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Revision History

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History	Effective Date
SESDPROC-102-R3, *Field Temperature Measurement*, replaces SESDPROC-102-R2 **Cover Page:** Author was changed from Ron Phelps to Hunter Johnson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Section 1.5:** In Section 1.5.1, added "The field investigators will" to the first sentence. Deleted Section 1.5.2. **Section 2:** In the first paragraph, kept the first sentence but replaced the rest of the paragraph with the following language for clarification purposes: "Temperature measurement devices such as pH, conductivity and dissolved oxygen (DO) meter thermistors will be verified against a National Institute of Standards and Technology (NIST)-traceable thermometer before each use as described in Section 3.2. Data Sonde thermistors will be verified on a monthly basis to ensure accuracy. These verification checks are documented in the instrument's tracking logbook." Deleted the second paragraph. **Section 3.1:** In the first paragraph, replaced the first sentence with: "Field temperature measurements may be made with a field thermometer, equipment thermistor, or NIST-traceable thermometer." In the second sentence, replaced "thermometer" with "temperature measurement device." **Section 3.2:** Created new sections 3.2.1 and 3.2.2 as follows: Converted the "Note" to new section 3.2.1. Replaced the first sentence with "Temperature measurement devices such as field thermometers and equipment thermistors will be verified against a NIST-traceable thermometer prior to use and should agree within ± 4.0°C." Converted the first paragraph to section 3.2.2. Revised the original language to clarify the verification requirements of the NIST-traceable thermometers that are used to verify other temperature measuring devices, such as field thermometers and equipment thermistors.	February 4, 2011

Section 3.3: Replaced "thermometer" with "temperature measurement device." **Section 3.4:** Added "if not in-situ" to number 4.	
SESDPROC-102-R2, *Field Temperature Measurement*, Replaces SESDPROC-102-R1 **Cover Page:** Author was changed from Marty Allen to Ron Phelps. **Revision History** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3** Changed Field Quality Manager to Document Control Coordinator. **Section 2** Deleted last sentence of paragraph 1. Deleted Fisher brand stem type in paragraph 2. **Section 3.1** Deleted "Normally" on the first sentence. Deleted the last sentence. **Section 3.2** Changed "Calibration" to "Verification." Added recalibration requirements.	June 13, 2008
SESDPROC-102-R1, *Field Temperature Measurement*, Replaces SESDPROC-102-R0 **General** Updated referenced procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 2** Added last paragraph regarding stopping measurements due to environmental conditions.	November 1, 2007
SESDPROC-102-R0, Field Temperature Measurement, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when measuring the temperature of aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring the temperature of aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain temperature measurements of the media being sampled, and that another method or measurement instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field log book and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting temperature measurements in the field. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The field investigators will address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

2 Quality Control

All thermometers should be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Temperature measurement devices such as pH, conductivity and dissolved oxygen (DO) meter thermistors will be verified against a National Institute of Standards and Technology (NIST)-traceable thermometer before each use as described in Section 3.2. Data Sonde thermistors will be verified on a monthly basis to ensure accuracy. These verification checks are documented in the instrument's tracking logbook.

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Temperature Measurement Procedures

3.1 General

Field temperature measurements may be made with a field thermometer, equipment thermistor, or NIST-traceable thermometer. At a minimum, the temperature measurement device should be capable of measuring in 0.1°C increments.

3.2 Instrument Verification

3.2.1 Field thermometers and thermistors

Temperature measurement devices such as field thermometers and equipment thermistors will be verified against a NIST-traceable thermometer prior to use and should agree within ± 4.0°C. Corrections may be applied for measurements up to ± 4.0°C depending on investigation objectives, but the instrument must be repaired or replaced beyond that range.

3.2.2 NIST-traceable thermometer

Verification of the NIST-traceable thermometers that are used to verify temperature measuring devices is accomplished by comparing temperature readings from the NIST-traceable thermometer to a thermometer that has an independent certification of accuracy traceable to the National Institute of Standards and Testing. Current certified thermometers are maintained by the SESD Analytical Support Branch and are called reference thermometers.

Each NIST-traceable thermometer is verified by comparing at least annually against a reference thermometer. If corrections need to be applied, they will be noted in the NIST-traceable thermometer. Depending on investigation objectives, project leaders may decide to apply the correction factor as necessary.

3.3 Inspections

All temperature measurement devices should be inspected for leaks, cracks, and/or function prior to each use.

3.4 Sample measurement procedures for thermometers/thermistors

(Make measurements in-situ when possible)

1. Clean the probe end with de-ionized water and immerse into sample.

2. If not measuring in-situ, swirl the instrument in the sample for mixing and equilibration.

3. Allow the instrument to equilibrate with the sample for at least one minute.

4. Suspend the instrument away from the sides and bottom, if not in-situ, to observe the temperature reading.

5. Record the reading in the log book. For most applications, report temperature readings to the nearest 0.5°C or to the nearest 0.1°C depending on need.

   Note: Always clean the thermometer with de-ionized water or a detergent solution, if appropriate, prior to storage and/or use.

3.5 Units

Degrees Celsius (°C) or Degrees Fahrenheit (°F)

Conversion Formulas:

°F = (9/5 °C) + 32 or °C = 5/9 (°F - 32)

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Turbidity Measurement**

Effective Date: January 29, 2013	**Number:** SESDPROC-103-R3

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Revision History

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History	Effective Date
SESDPROC-103-R3, *Field Turbidity Measurement*, replaces SESDPROC-103-R2 **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Author was changed from Ron Phelps to Timothy Simpson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Danny France. The FQM was changed from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Section 1.5.1:** Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used. **Section 2:** In the first paragraph, replaced "and" in the second sentence with "or". **Section 3.2:** Replaced "Meter" with "Instrument" in section title. Replaced the first paragraph with the following language: "Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within ± 10 % of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:" Removed section on calibration and verification of the HACH 2100P Turbidimeter. Added Section 3.2.1, Meter Calibration and Verification, that includes information on the calibration and verification of the 2100Q Turbidimeter. Replaced Section 3.3, Probe Calibration and Verification, with Section 3.2.1, Probe Calibration and Verification.	January 29, 2013

Section 3.3: Added "Depending on the meter, the sample measurement procedure can differ slightly." Converted Section 3.3 into Section 3.3.1, Grab Sample Measurement, and Section 3.3.2, *In-Situ* Measurement. **Section 3.3.1:** In Item #3 replaced "Press I/O and the instrument will turn on." with "Turn instrument on." In Item #5 and #6 added "If appropriate". In Item #8 added "or rinse out with sample water prior to the next reading". **Section 3.5:** Replaced the section with the following language: "Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed. While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by ± 10 % (depending on the application) from the calibration standard, the meter must be re-calibrated. A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook."	
SESDPROC-103-R2, *Field Turbidity Measurement*, replaces SESDPROC-103-R1 **Cover Page:** Author was changed from Marty Allen to Ron Phelps. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3:** Changed Field Quality Manager to Document Control Coordinator.	June 13, 2008

SESDPROC-103-R1, *Field Turbidity Measurement*, replaces SESDPROC-103-R0 **General** Deleted all references to SOSA. Updated referenced procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 2** Added last paragraph regarding stopping measurements due to environmental conditions. **Section 3.5** Re-phrased operational check 2 for clarity.	November 1, 2007
SESDPROC-103-R0, Field Turbidity Measurement, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when conducting field turbidity measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring turbidity of various, aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain turbidity measurements of the media being sampled, and that another method or turbidity measurement instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field turbidity measurements. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field turbidity measurements pertinent to the sampling event should be recorded in the field logbook for the event. All records should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010).

2 Quality Control

All turbidity meters and probes shall be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter or probe is taken to the field, it shall be properly calibrated or verified, according to Sections 3.2 and 3.3 of this procedure, to ensure it is operating properly. These calibration and verification checks shall be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Turbidity Measurement Procedures

3.1 General

Turbidity is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Many methods are available for the measurement of turbidity including turbidimeters and optical probes. Turbidity is measured by determining the amount of scatter when a light is passed through a sample.

3.2 Instrument Calibration and Verification

Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within ± 10 % of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:

3.2.1 Meter Calibration and Verification

HACH 2100Q Turbidimeter:

Portable turbidimeters are calibrated with Formazin Primary Standards. The manufacturer recommends calibration with a primary standard such as StablCal® Stabilized Standards or with formazin standards every three months.

Generally only a calibration verification measurement is required in the field; however, if a calibration is needed, record a post calibration reading for each calibration standard used.

Meter Verification:

1. Push **Verify Cal** to enter the Verify menu.

2. Gently invert the liquid standard several times prior to insertion into meter. Insert the 10.0 NTU (or other defined value) Verification Standard and close the Lid.

3. Push **Read**. The display shows "Stabilizing" and then shows the result and tolerance range.

4. Push **Done** to return to the reading display. Repeat the calibration verification if the verification failed. If a meter is unable to pass verification, then that meter will need to be calibrated.

Meter Calibration:

1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display. **Note:** Gently invert each standard several times before inserting the standard and use a non-abrasive, lint-free paper or cloth to wipe off the standards.

2. Insert the 20 NTU StablCal Standard and close the lid. Push **Read**. The display shows "Stabilizing" and then shows the result. Record the result.

3. Repeat Step 2 with the 100 NTU and 800 NTU StablCal Standard. Record both results.

4. Push **Done** to review the calibration details.

5. Push **Store** to save the results. After a calibration is complete, the meter automatically goes into the Verify Cal mode.

3.2.2 Probe Calibration and Verification

The manufacturer's instruction manual should be consulted for specific procedures regarding probe's calibration, maintenance and use. Their calibration must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. The following are basic guidelines for calibration/verification of probes and are provided as an example:

1. Turn the meter "ON" and allow it to stabilize

2. Immerse the probe in the first standard solution and calibrate the probe against the solution.

3. Rinse the probe with de-ionized water, remove excess rinse water and calibrate the probe using additional standards as appropriate.

4. Record the standard values used to calibrate the meter.

3.3 Sample Measurement Procedures

Depending on the meter, the sample measurement procedure can differ slightly.

3.3.1 Grab Sample Measurement

These procedures should be followed when conducting turbidity measurements of grab samples:

1. Collect a representative sample and pour off enough to fill the cell to the fill line (about 15 mL) and replace the cap on the cell.

2. Wipe off excess water and any streaks with a soft, lint-free cloth (lens paper).

3. Turn instrument on. Place the meter on a flat, sturdy surface. Do not hold the instrument while making measurements.

4. Insert the sample cell in the instrument so the diamond or orientation mark aligns with the raised orientation mark in the front of the cell compartment. Close the lid.

5. If appropriate, select manual or automatic range selection by pressing the range key.

6. If appropriate, select signal averaging mode by pressing the Signal Average key. Use signal average mode if the sample causes a noisy signal (display changes constantly).

7. Press Read. The display will show ---- NTU. Then the turbidity is displayed in NTU. Record the result after the lamp symbol turns off.

8. Rinse the cell with de-ionized water or rinse out with sample water prior to the next reading.

3.3.2 In-Situ Measurement

These procedures should be followed when conducting in-situ turbidity measurements:

1. Place the probe into the media to be measured and allow the turbidity reading to stabilize. Once the reading has stabilized, record the measurement in the logbook.

2. When deploying meters for extended periods of time, ensure the measurement location is representative of average media conditions.

3.5 Operational check

Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.

While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by ± 10 % (depending on the application) from the calibration standard, the meter must be re-calibrated.

A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

3.6 Units

Turbidity measurements are reported in nephelometric turbidity units (NTUs). It is important to note that if the turbidity measurements are for NPDES reporting purposes, all values above 40 NTU must be diluted with turbidity free-water and calculated by multiplying by a dilution factor.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Turbidity Measurement**

Effective Date: January 29, 2013	**Number:** SESDPROC-103-R3

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This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-103-R3, *Field Turbidity Measurement*, replaces SESDPROC-103-R2 **General:** Corrected any typographical, grammatical and/or editorial errors. **Cover Page:** The Author was changed from Ron Phelps to Timothy Simpson. The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Danny France. The FQM was changed from Laura Ackerman to Bobby Lewis. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Section 1.5.1:** Updated the SHEMP Manual reference to reflect that the most recent version of the Manual will be used. **Section 2:** In the first paragraph, replaced "and" in the second sentence with "or". **Section 3.2:** Replaced "Meter" with "Instrument" in section title. Replaced the first paragraph with the following language: "Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within ± 10 % of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:" Removed section on calibration and verification of the HACH 2100P Turbidimeter. Added Section 3.2.1, Meter Calibration and Verification, that includes information on the calibration and verification of the 2100Q Turbidimeter. Replaced Section 3.3, Probe Calibration and Verification, with Section 3.2.1, Probe Calibration and Verification.	January 29, 2013

Section 3.3: Added "Depending on the meter, the sample measurement procedure can differ slightly." Converted Section 3.3 into Section 3.3.1, Grab Sample Measurement, and Section 3.3.2, *In-Situ* Measurement. **Section 3.3.1:** In Item #3 replaced "Press I/O and the instrument will turn on." with "Turn instrument on." In Item #5 and #6 added "If appropriate". In Item #8 added "or rinse out with sample water prior to the next reading". **Section 3.5:** Replaced the section with the following language: "Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed. While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by ± 10 % (depending on the application) from the calibration standard, the meter must be re-calibrated. A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook."	
SESDPROC-103-R2, *Field Turbidity Measurement*, replaces SESDPROC-103-R1 **Cover Page:** Author was changed from Marty Allen to Ron Phelps. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3:** Changed Field Quality Manager to Document Control Coordinator.	June 13, 2008

SESDPROC-103-R1, *Field Turbidity Measurement*, replaces SESDPROC-103-R0 **General** Deleted all references to SOSA. Updated referenced procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 2** Added last paragraph regarding stopping measurements due to environmental conditions. **Section 3.5** Re-phrased operational check 2 for clarity.	November 1, 2007
SESDPROC-103-R0, Field Turbidity Measurement, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when conducting field turbidity measurements in aqueous phase environmental media, including groundwater, surface water and certain wastewaters.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring turbidity of various, aqueous phase environmental media in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain turbidity measurements of the media being sampled, and that another method or turbidity measurement instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when conducting field turbidity measurements. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Manual (Most Recent Version) and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

All field turbidity measurements pertinent to the sampling event should be recorded in the field logbook for the event. All records should be entered according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010).

2 Quality Control

All turbidity meters and probes shall be maintained and operated in accordance with the manufacturer's instructions and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Before a meter or probe is taken to the field, it shall be properly calibrated or verified, according to Sections 3.2 and 3.3 of this procedure, to ensure it is operating properly. These calibration and verification checks shall be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to insure the instrument is operated within the manufacturer's specified range of operating temperatures. For instruments that are deployed for in-situ measurements, the temperature of the medium being monitored should be measured and recorded in the logbook prior to deployment. *In-situ monitoring equipment may be utilized in unattended deployments where autonomous logging may preclude temperature measurement prior to deployment. Because in situ instrumentation generally has a wide range of operating temperature, the field investigator may utilize professional judgment in determining if the operating environment is suitable for unattended deployment.*

If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Turbidity Measurement Procedures

3.1 General

Turbidity is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Many methods are available for the measurement of turbidity including turbidimeters and optical probes. Turbidity is measured by determining the amount of scatter when a light is passed through a sample.

3.2 Instrument Calibration and Verification

Many brands of instruments are commercially available for the measurement of turbidity incorporating a wide variety of technologies. The manufacturer's instruction manual should be consulted for specific procedures regarding their calibration, maintenance and use. Calibration of any measurement instrument must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. Depending on the instrument, the verification and calibration can differ slightly. If the instrument readings do not agree within ± 10 % of the calibration standards, the unit must be recalibrated, repaired or replaced. The following are basic guidelines for calibration/verification of meters and are provided as an example:

3.2.1 Meter Calibration and Verification

HACH 2100Q Turbidimeter:

Portable turbidimeters are calibrated with Formazin Primary Standards. The manufacturer recommends calibration with a primary standard such as StablCal® Stabilized Standards or with formazin standards every three months.

Generally only a calibration verification measurement is required in the field; however, if a calibration is needed, record a post calibration reading for each calibration standard used.

Meter Verification:

1. Push **Verify Cal** to enter the Verify menu.

2. Gently invert the liquid standard several times prior to insertion into meter. Insert the 10.0 NTU (or other defined value) Verification Standard and close the Lid.

3. Push **Read**. The display shows "Stabilizing" and then shows the result and tolerance range.

4. Push **Done** to return to the reading display. Repeat the calibration verification if the verification failed. If a meter is unable to pass verification, then that meter will need to be calibrated.

Meter Calibration:

1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display. **Note:** Gently invert each standard several times before inserting the standard and use a non-abrasive, lint-free paper or cloth to wipe off the standards.

2. Insert the 20 NTU StablCal Standard and close the lid. Push **Read**. The display shows "Stabilizing" and then shows the result. Record the result.

3. Repeat Step 2 with the 100 NTU and 800 NTU StablCal Standard. Record both results.

4. Push **Done** to review the calibration details.

5. Push **Store** to save the results. After a calibration is complete, the meter automatically goes into the Verify Cal mode.

3.2.2 Probe Calibration and Verification

The manufacturer's instruction manual should be consulted for specific procedures regarding probe's calibration, maintenance and use. Their calibration must be conducted and/or verified prior to each use or on a daily basis, whichever is most appropriate. The following are basic guidelines for calibration/verification of probes and are provided as an example:

1. Turn the meter "ON" and allow it to stabilize

2. Immerse the probe in the first standard solution and calibrate the probe against the solution.

3. Rinse the probe with de-ionized water, remove excess rinse water and calibrate the probe using additional standards as appropriate.

4. Record the standard values used to calibrate the meter.

3.3 Sample Measurement Procedures

Depending on the meter, the sample measurement procedure can differ slightly.

3.3.1 Grab Sample Measurement

These procedures should be followed when conducting turbidity measurements of grab samples:

1. Collect a representative sample and pour off enough to fill the cell to the fill line (about 15 mL) and replace the cap on the cell.

2. Wipe off excess water and any streaks with a soft, lint-free cloth (lens paper).

3. Turn instrument on. Place the meter on a flat, sturdy surface. Do not hold the instrument while making measurements.

4. Insert the sample cell in the instrument so the diamond or orientation mark aligns with the raised orientation mark in the front of the cell compartment. Close the lid.

5. If appropriate, select manual or automatic range selection by pressing the range key.

6. If appropriate, select signal averaging mode by pressing the Signal Average key. Use signal average mode if the sample causes a noisy signal (display changes constantly).

7. Press Read. The display will show ---- NTU. Then the turbidity is displayed in NTU. Record the result after the lamp symbol turns off.

8. Rinse the cell with de-ionized water or rinse out with sample water prior to the next reading.

3.3.2 In-Situ Measurement

These procedures should be followed when conducting in-situ turbidity measurements:

1. Place the probe into the media to be measured and allow the turbidity reading to stabilize. Once the reading has stabilized, record the measurement in the logbook.

2. When deploying meters for extended periods of time, ensure the measurement location is representative of average media conditions.

3.5 Operational check

Even though it is not necessary to re-calibrate turbidity meters at regular intervals during the day, depending on the instrument, it may be appropriate to occasionally perform operational checks to determine if site conditions, such as an increase in temperature, have impacted the meter's performance. If an operational check is warranted, the following procedure should be followed to ensure that the performance of the meter has not changed.

While in use, periodically check the turbidity by rinsing the probe with de-ionized water, blot dry or otherwise remove excess rinse water and immerse it into the appropriate calibration standard. If the measured turbidity differs by ± 10 % (depending on the application) from the calibration standard, the meter must be re-calibrated.

A post-operation instrument verification check will be performed using the appropriate standard(s) at the end of the day or after all measurements have been taken for a particular period of operation. These measurements must be recorded in the field logbook.

3.6 Units

Turbidity measurements are reported in nephelometric turbidity units (NTUs). It is important to note that if the turbidity measurements are for NPDES reporting purposes, all values above 40 NTU must be diluted with turbidity free-water and calculated by multiplying by a dilution factor.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Global Positioning System**

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Revision History

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History	Effective Date
SESDPROC-110-R3, *Global Positioning System*, replaces SESDPROC-110-R2 **Cover Page:** The EIB Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. Changed Field Quality Manager to Document Control Coordinator.	April 20, 2011
SESDPROC-110-R2, *Global Positioning System*, replaces SESDPROC-110-R01 **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove‟s title from Acting Chief to Chief. **Section 1.4** Alphabetized and added a reference.	November 1, 2007
SESDPROC-110-R1, *Global Positioning System*, replaces SESDPROC-110-R0 This revision reflects the following changes: **General** Extensive rewrite with additional information added to most sections. Brian Striggow added as coauthor. **Section 2.1** Rewritten description of GPS system. Descriptions of "GPS Accuracy Factors" and "Differential GPS" isolated as separate subsections.	October 1, 2007

Section 2.2 This Section, titled "Requirements for Locational Information" inserted. Supplies guidance on accuracy requirements for various study types and potential means used to obtain requisite accuracy. Subsection on datums and data formats included. **Section 2.3** "Quality Control Procedures", previously numbered as Section 2.2. Most information in this section moved to Section 2.1 discussion of Accuracy Factors and Section 2.4 discussion of specific receivers. Explicit statement added that no specific GPS quality control procedures are required for most SESD studies. **Section 2.4** Information added to sections on Trimble and Garmin receivers. **Section 2.5** Issue of electronic data-logging addressed. Storage of electronic data records addressed.	
SESDPROC-110-R0, Global Positioning System, Original Issue	March 22, 2007

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Contents

1 General Information

1.1 Purpose

This document describes the Global Positioning System (GPS) and procedures, methods and considerations to be used and observed when using GPS to record location data in the field. Guidance is provided on accuracy requirements for various uses of location data and potential means to obtain the requisite accuracy. This document contains direction developed solely to provide internal guidance to SESD employees.

1.2 Scope/Application

The procedures contained in this document are to be used by SESD field investigators when using the Global Positioning System to obtain the geographical coordinates of sampling locations and/or measurements during field investigations. In SESD investigations, GPS is the preferred means of collecting horizontal location information. In most cases the accuracy of GPS is unsuitable for collection of elevation data.

On the occasion that SESD field personnel determine that any of the procedures described in this section cannot be used to obtain the required coordinate information and alternate procedures are employed, the alternate procedure will be documented in the field log book, along with a description of the circumstances requiring its use. GPS users must be currently qualified as proficient in the operation of the specific GPS equipment to be used. The manufacturer"s operation manuals should be used for detailed information on the use of specific GPS equipment. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Rand Corporation, The Global Positioning System, Assessing National Policies, Appendix B, GPS History, Chronology, and Budgets, 1995.

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version.

Trimble® Navigation Limited, Mapping Systems General Reference, Revision B, 1996.

USEPA, Global Position Systems – Technical Implementation Guidance, Office of Environmental Information (EPA/250/R-03/001), 2003.

USEPA, GIS Technical Memorandum 3. Global Positioning Systems – Technology and It"s Application in Environmental Programs, Research and Development (PM-225). EPA/600/R-92/036, 1992.

USEPA, Locational Data Policy, Office of Information Resources Management, IRM Policy Manual 2100 Chapter 13, 1991.

2 Methodology

2.1 General

2.1.1 GPS Description

The Navigation Satellite Time and Ranging (NAVSTAR) Global Positioning System (GPS) is a worldwide radio-navigation system created by the U. S. Department of Defense (DOD) to provide navigation, location, and timing information for military operations. System testing using a limited number of satellites began in 1978 with the system being declared fully operational in 1995. The system was declared available for civilian uses in the 1980s and has seen burgeoning civilian application for navigation and mapping.

The GPS system consists of three basic elements: the space segment, control segment, and user segment. The space segment consists of the constellation of up to 24 active NAVSTAR satellites in six orbital tracks. The satellites are not in geo-synchronous orbit and are in constant motion relative to a ground user. The control segment consists of several ground stations that serve as uplinks to the satellites and that make adjustments to satellite orbits and clocks when necessary. The user segment consists of the GPS receiver which will typically consist of an antenna, multi-channel receiver, and processing unit.

For the purposes of this document, the user segment GPS receivers may be loosely grouped into Recreational and Navigational receivers (henceforth referred to as General Use receivers), Mapping Grade receivers, and Survey Grade receivers.

- Most General Use grade receivers are available on the retail market to consumers for a variety of applications including boating, hiking, and automotive navigation. They display an instantaneous reading of position and are generally not optimized for data collection. Waypoints containing instantaneous position fixes can often be stored and downloaded. The accuracy of these receivers is adequate for many environmental applications.

- Mapping Grade receivers are used for applications such as resource management and Geographical Information System (GIS) feature collection. The receivers are capable of averaging multiple position fixes for greater accuracy and then data-logging the results with sufficient information to post-correct the positions as described below. The potential accuracy that can be achieved may be better than one meter.

- Survey Grade receivers can provide accuracy at the centimeter level by using long occupation times and special techniques for receiver use and data processing. Survey Grade receivers are not used by SESD in field investigations.

GPS receivers derive positions by simultaneously measuring the distance (range) to several satellites in precisely known orbits, and using triangulation of the ranges to calculate a unique position for the receiver. The range to each satellite is determined by precisely measuring the transit time of radio signals broadcast from the satellites.

2.1.2 GPS Accuracy Factors

The accuracy of the basic GPS system is approximately 15m. GPS accuracy can be affected by a number of factors including the Selective Availability feature, atmospheric delays, satellite clock and orbit errors, multipath signals, signal strength, and satellite geometry relative to the user.

In the early GPS implementation, the DOD used a feature known as Selective Availability (SA) to degrade the quality and subsequent accuracy of the GPS signals to non-DOD users. With Selective Availability enabled, accuracy of position fixes could be as poor as 100m without the use of differential correction techniques described below. Currently there is no SA limitation in accuracy in place with a stated Executive Branch intention to not return to the use of the SA signal degradation.

As satellites move in their orbits and some signals are blocked by obstructions, the geometry of the available satellite signals relative to the user will constantly change. When the satellites with available signals are clustered closely together in the sky, small errors in range will result in large errors in reported position. Conversely, when the satellites are distributed more broadly across the sky, the resultant position errors will be at their minimum. The general measure of this phenomenon is Dilution of Precision (DOP), which may be represented as Position Dilution of Precision (PDOP), or more specifically for geographical coordinate collection, Horizontal Dilution of Precision (HDOP). Mapping and Survey Grade receivers generally can calculate and display DOP and allow the user to limit logging to times when the higher potential accuracy conditions of low DOP prevail. General Use receivers may display DOP and use DOP with other factors to estimate a general accuracy figure. DOP may range from approximately 2 to 50, with high quality work usually requiring a HDOP of less than 4-6.

Signal strength and multipath signals relate to the strength and quality of the signal reaching the receiver antenna. Signal attenuation by the atmosphere, buildings, and tree cover limit the accuracy of the ranges obtained. The measure of signal strength is Signal to Noise Ratio (SNR), generally measured in decibels(db). Most receivers of any grade will display the SNR of the satellite signals in a bar graph or table. Mapping Grade Receivers generally allow the user to specify a minimum signal strength for the use of a satellite signal (commonly 2-15db). Poor signal strength can be resolved by waiting for satellite locations to change or moving the receiver location. Multipath signals result

from portions of the satellite signal bouncing off terrain, structures, or atmospheric disturbances, resulting in a degraded total signal. Higher quality Mapping Grade receivers may be capable of rejecting the stray multipath signals, such as Trimble® receivers using Everest™ technology.

2.1.3 Differential GPS

Selective Availability, clock errors, and orbital errors affect all GPS users and atmospheric delays affect all users over a relatively wide region. A second GPS receiver in the same general area as the user will experience the same errors from these sources as the user‟s receiver. Consequently, correction factors from a remote station at a known location can be applied to the user‟s receiver in a process known as Differential GPS (DGPS). DGPS can be applied in real-time using additional radio signals, or after the collection event by a method called post-correction.

Real-time DGPS uses established networks of base stations at precisely surveyed locations. The US Coast Guard operates a system of 80 base stations which became fully operational in 1999. The range corrections are broadcast on marine radiobeacon frequencies, with redundant coverage of most of the US coastline and the Mississippi River. There is near complete single beacon coverage of most of the internal US, but there are known gaps in coverage in both EPA Region 4 and the US as a whole. The system is sometimes referred to using the more general term DGPS or in nomenclature referring to the beacon-based nature of the system. Beacon-based DGPS is implemented primarily in Navigational and Mapping Grade receivers.

Real-time DGPS can also be implemented with a Space Based Augmentation System (SBAS). The most common SBAS currently used in the United States is the Wide Area Augmentation System (WAAS), developed by the Federal Aviation Administration to meet the additional demands on GPS for aircraft navigation. The WAAS network of base stations collects information on satellite clock errors, orbital errors, and atmospheric conditions. The error information is transferred to satellites in geo-synchronous orbits and subsequently broadcast to suitably equipped GPS receivers on frequencies compatible with the GPS range signals. While beacon-based DGPS passes range corrections to the receivers, WAAS communicates a model for the errors which is capable of providing more accurate corrections. Current Mapping Grade receivers will likely use WAAS with or without the option of beacon-based DGPS. All but the least expensive General Use receivers are generally equipped with WAAS differential correction capability.

Post-Corrected DGPS is generally accomplished by downloading the receiver survey files to a desktop or laptop computer and then retrieving correction files for the same time period (generally via the internet) from an established base station in the area of the survey. Post-processed accuracy improves with proximity of the base station to the surveyed locations and base station data should be used from a station within 300km of the site surveyed. The survey positions are processed by application software and a new

set of positions is generated using the correction data. The capability for post-processed differential correction is generally limited to Mapping Grade and Survey Grade receivers.

Various factors limit GPS accuracy in the vertical plane to approximately half of that obtainable in the horizontal plane, i.e., if a location fix is accurate to 3 m in the horizontal plane, it may only be accurate to 6 m in the vertical plane. Since relatively high accuracy is usually required for the uses of elevation data, GPS is rarely used to obtain and report elevations.

2.2 Requirements for Locational Information

2.2.1 Data Uses

Locational information can serve many purposes in an environmental investigation, a few of which are listed below:

1. Providing an unambiguous means to identify facilities or sampling plats.
2. Providing locational information to key analytical data in a GIS based data archiving system to the original sampling locations.
3. Differentiating watersheds.
4. Providing information to calculate extents and volumes of contamination.
5. Providing a means to relocate the media represented by samples for removal or treatment.
6. Providing information to prepare presentation graphics of sampling locations.

Depending on the specific uses for the data and the type of work being performed, there will be different needs for the accuracy of the locational data. Studies where a sample represents a large area of relatively homogeneous material would not require the same accuracy as the location of a permanent monitoring well. Below are broad guidelines for the accuracy that might be required for different applications.

Desired Accuracy	**Application**
100 m	Open ocean work where sample is presumed to be representative of a large area
20 m	Open water work (lakes or estuaries) where sample is presumed to be representative of a large area
10 m	Stream and river work where samples are presumed to be broadly representative of a reach
5-3 m	Stream work where samples are representative of a specific narrowly defined section

10 m	Air Monitoring Stations
10 - 3 m	Microscale air monitoring
3 - 1 m	Permanent monitoring wells
1 m	Locations of 'Hot Spots' destined for removal of limited areal extent
3 - 1 m	Locations of Temporary groundwater wells in plumes requiring narrow delineation
3 m	Locations of Temporary groundwater wells in broad plumes
3 m	Locations of environmental samples with sample spacing >20 m
5 m	Locations of environmental samples with sample spacing >60 m
200 - 20 m	Coordinates describing a facility where mobile waste units are sampled
30 - 3 m	Locations of industrial process areas or NPDES permitted facilities where the sampling locations are described in field notes relative to the process or site features

Specific demands of a study may drive increased or decreased requirements for accuracy. The preferred means of locational data collection for most studies will be GPS, although alternate means are permissible if they meet accuracy requirements. The following table indicates the accuracy that may be expected from various means of establishing coordinates.

Accuracy	**Description**
200 - 50 m	Map Derived, coarse work
40 - 20 m	Map Derived, fine work or using GIS with digital imagery
15 m	General Use Grade GPS, w/o WAAS
5 m	General Use Grade GPS, w/ WAAS or beacon corrections
10 m	Mapping Grade GPS, no corrections, averaged readings,
3 m	Mapping Grade GPS w/ differential correction, averaged readings
1 m	Mapping Grade GPS w/ differential correction, controlled DOP and SNR, averaged readings
10 cm	Surveying Grade GPS or optical surveying (dependent on baseline length)

Accuracy is a term used to describe the degree of conformity of a measurement. In GPS, accuracy is usually specified as an estimate of the radius from the measured coordinates that is likely to include the actual coordinates. The estimate is based on several standard deviations probability of including the actual point in the measurement. As such, it is recognized that some measurements will fall outside of the specified accuracy. For the purposes of SESD GPS work, the nominal accuracy figures derived from manufacturer‟s literature for specific operating conditions, displayed by the receiver at the time of feature collection, or output from processing software will be taken at face value.

2.2.2 Datums and Data formats

In general, a datum is a reference from which other measurements are taken. In the development of surveying systems by civil entities, different datums were used as base references that will result in differing coordinates for the same location. A GPS receiver will generally display coordinates in a number of different user-selected datums. **Unless there are specific requirements on a project, all SESD work should be conducted using the WGS84 datum. A**lternatively, the nearly equivalent NAD83 datum may be used if WGS84 is unavailable as a receiver option. If an alternate coordinate system is used where coordinates are obtained and recorded in field logbooks, the use of the alternate coordinate system should also be noted in the logbook.

The SESD Data Archival and Retrieval System (DART) requires that coordinates for sample locations be entered in the WGS84 datum and dd.dddddd format. Unless specific project requirements dictate otherwise, all coordinates explicitly stated in reports will be in WGS84 format and in all cases the datum used will be specified.

There is no SESD policy on significant digits for GPS information, and accuracy should not be implied from the presence of significant digits in reported coordinates. However, good scientific practice should be followed in the presentation of locational information in order that useful information not be truncated or a higher degree of accuracy implied. The following table shows the incremental distance in latitude represented by the least significant digit for various coordinate formats:

dd.dddddd°	Approximately 4" or 10 cm
dd.ddddd°	Approximately 44" or 1.1 m
dd.dddd°	Approximately 36' or 11 m
dd°mm'ss"	Approximately 100' or 30 m
dd°mm'ss.x"	Approximately 10' or 3 m
dd°mm'ss.xx"	Approximately 1' or 30 cm
dd°mm.xxxx'	Approximately 7" or 18 cm
dd°mm.xxx'	Approximately 6' or 1.8 m
dd°mm.xx'	Approximately 60' or 18 m

2.3 Quality Control Procedures

By nature of its origin in the DOD and recent application to aircraft navigation, the GPS is designed for high reliability. GPS failures resulting in an incorrect reading beyond the bounds of known errors are so rare that the possibility can be ignored for most SESD studies. If a study requires the verification of receiver function, this can be accomplished by verifying that a receiver displays the correct position while occupying a known benchmark.

2.4 Special Considerations

The application will dictate the type of receiver used. There are several specific considerations for the use of the various receiver types.

2.4.1 Special considerations for the use of Trimble® Mapping Grade Receivers

The suggested settings for Trimble® receivers are:

1. Position mode: Overdetermined 3D (or manual 3D if only 4 satellites are visible)
2. Elevation mask: 15 degrees
3. PDOP: 6
4. Signal to noise ratio (SNR) mask: 6
5. Dynamics code: Land
6. Logging interval: 1 second for points, 5 seconds for lines and areas
7. Audible click: Yes
8. Log DOP data: Yes
9. Coordinate System: Geographic Datum WGS84

When using Trimble® Geoexplorer receivers, the option of using the accuracy „slider bar‟ display may be used to consolidate many of the above parameters. The slider bar position must be positioned at midscale or towards the „Precision‟ end of the scale to achieve 1m accuracy work. When conducting work requiring less accuracy, the slider should be initially positioned midscale and may be adjusted toward the Production‟ end of the scale if the higher level of accuracy can not be maintained.

The receivers may be configured to force real-time position correction. The use of real-time correction reduces the risk of being unable to obtain appropriate post-correction files, but may limit productivity when differential signals cannot be

received or are intermittent. The use of forced real-time vs. post-correction is an operator decision. If the positions are not to be downloaded and the accuracy of differential correction is required, it will be necessary to force real-time correction.

Trimble® receivers at SESD contain a data dictionary that can facilitate the management of GIS data. If the COC_GIS dictionary is selected at the time of file creation, SESD standard media codes can be assigned to features at the time of logging that will accompany the data through the download process. The use of the COC_GIS data dictionary can simplify the management of the data when processed in a GIS system or when entered into the DART system.

The logging interval of most Trimble® GPS receivers defaults to a 5 second interval. This may be changed to a 1 second interval to expedite feature collection. A point feature should have a minimum of 36 positions logged to obtain the additional accuracy afforded by averaging positions. After a minimum of 36 positions are logged and the feature is closed, the averaged position can be obtained by selecting the feature on the „Map" screen. The averaged position should always be the one entered into field notebooks.

Some Trimble® receivers may only display data in dd°mm"ss.sss" format. If the coordinates are downloaded and processed through Pathfinder Office software, they can be output in the dd.dddddd (or any other) format. If the coordinates were only recorded in field logbooks, they can be converted to decimal degrees as follows:

Converting to decimal degrees (dd.dddddd) from degrees°minutes"seconds" (dd°mm"ss.sss"):

dd.dddddd = dd + (mm/60) + (ss.sss/3600)

Example: Convert 33°28"45.241" to decimal degrees

33 + (28/60) + (45.241/3600) = 33.479236

The reverse conversion is accomplished as follows:

Converting to degrees°minutes"seconds" from decimal degrees

Starting with dd.dddddd

Multiply .dddddd by 60 to obtain mm.mmmm

Multiply .mmmm by 60 to obtain ss.sss

Then dd°mm"ss.sss" = dd & mm & ss.sss

Example: Convert 33.479236 to dd°mm‟ss.sss” format

Multiply .479236 by 60 to obtain 28.7540 (mm.mmmm)

Multiply .7540 by 60 to obtain 45.241 (ss.sss)

Dd°mm‟ss.sss” = 33° & 28‟ & 45.241” = 33°28‟45.241”

2.4.2 Special considerations for the use of Garmin® General Use Grade Receivers

The standard format for navigational purposes is decimal minutes (dd°mm.mmm‟). This format is utilized due to the fact that nautical navigation charts are set up in this format. However, location information must be converted to a decimal degree (dd.ddddd°) format in order for GIS software to properly interpret the information. The conversion is accomplished by dividing the minutes portion of the coordinates by 60.

<u>Converting to decimal degrees from decimal minutes:</u>

dd.ddddd° = dd + (mm.mmm/60)

Example: Convert 81°49.386 degrees to decimal degrees

81 + (49.386/60) = 81.8231 degrees

<u>The reverse conversion is accomplished as follows:</u>

dd°mm.mmm‟ = dd & (.ddddd*60)

Example: Convert 81.8231 degrees to decimal minutes (dd°mm.mmm‟)

Multiply .8231 by 60 to obtain 49.386 (mm.mmm)

81° & 49.386‟ = 81°49.386‟

GPS users need to familiarize themselves with the differences between the two formats, as they appear similar. Spreadsheets can automate the conversion process.

2.5 Records

The GPS coordinates and the SESD equipment identification number of the GPS receiver should be recorded in field logbooks at the time of GPS coordinate collection. The data logging capability of receivers may be used in lieu of the requirement to record the coordinates in logbooks when the following conditions can be met:

1. The location can easily be found later if it needs to be resurveyed prior to demobilization. A permanent monitoring well can easily be resurveyed, while most open-water work would not afford this opportunity.

2. The data is downloaded and ascertained to meet the accuracy requirements for the project prior to demobilization from the site.

3. The data is stored in at least two separate locations for transport, such as a laptop hard drive and a flash drive or compact disc.

In all cases where positions are electronically recorded, the provisions of the Electronic Records section of the SESD Operating Procedure for Control of Records (SESDPROC-002) should be followed.

Where locational data is collected and processed electronically, but not reported explicitly in the final report, a copy of the coordinates in text format should be output and entered into the project file in paper or electronic form. The output should include:

1. Latitude, generally in dd.dddddd format.

2. Longitude, generally in dd.dddddd format.

3. Date of collection.

4. DOP information where it supports the accuracy requirements.

5. Correction status of each point where it supports the accuracy requirements.

6. The datum used for the export.

Trimble® Pathfinder Office will create files with this information by exporting to a text file. The information will be contained in the .pos and .inf files.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Groundwater Level and Well Depth Measurement**

Effective Date: January 29, 2013	**Number:** SESDPROC-105-R2

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

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SESDPROC-105-R2, *Groundwater Level and Well Depth Measurement*, replaces SESDPROC-105-R1 **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the Author from Don Hunter to Brian Striggow Changed the EIB Chief from Antonio Quinones to Danny France. Changed the FQM from Laura Ackerman to Bobby Lewis. **Revision History:** In the 3rd sentence, changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted reference to the H: drive. Changed Field Quality Manager to Document Control Coordinator. **Section 1.4:** Updated references. **Section 2, 1st bullet:** Replaced references to "calibration" of groundwater level measuring devices to "verification" of such devices. In the 1st sentence, updated the language to reflect that devices used to measure groundwater levels will be verified annually against a NIST-traceable measuring tape instead of an Invar® steel surveyor's chain. In the 2nd sentence, added "…with an allowable error of 0.03 feet in the first 30 feet." Added the last sentence (reference to SESDFORM-043, *Well Sounder Function Check and Verification* form). **Section 3.1, 1st paragraph, last sentence:** Rewrote the sentence for clarity. Added the 4th paragraph regarding equilibration of water levels. **Added new Section 3.3,** *Special Considerations for Water Level Measurements at Low Groundwater Gradient.* Re-numbered following sub-sections. **Section 3.4:** Added the 5th sentence and the last sentence.	January 29, 2013

SESDPROC-105-R1, *Groundwater Level and Well Depth Measurement*, replaces SESDPROC-105-R0 **General** Updated referenced procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Alphabetized and revised the referencing style for consistency.	November 1, 2007
SESDPROC-105-R0, Groundwater Level and Well Depth Measurement, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when determining water levels and depths of wells.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators to measure water levels and depths of wells. On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used for water level or depth determination, the variant procedure(s) will be documented in the field logbook and the subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network. The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when measuring water levels in wells and determining their depths. Refer to the SESD Safety, Health and

Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when measuring water levels and depths of wells:

- Special care must be taken to minimize the risk of cross-contamination between wells when conducting water level and depth measurements. This is accomplished primarily by decontaminating the sounders or other measuring devices between wells, according to SESD Operating Procedure for Field Equipment Cleaning and Decontamination, (SESDPROC-205) and maintaining the sounders in clean environment while in transit between wells.
- Water levels and well depths measured according to these procedures should be recorded in a bound logbook dedicated to the project as per SESD Operating Procedure for Logbooks (SESDPROC-010). Serial numbers, property numbers or other unique identification for the water level indicator or sounder must also be recorded.

2 Quality Control Issues

There are several specific quality control issues pertinent to conducting water level and depth measurements at wells. These are:

- Devices used to measure groundwater levels will be verified annually against a National Institute of Standards and Technology (NIST) traceable measuring tape.. These devices should check to within 0.01 feet per 10 feet of length with an allowable error of 0.03 feet in the first 30 feet. Before each use, these devices should be prepared according to the manufacturer's instructions (if appropriate) and checked for obvious damage. All verification and maintenance data should be documented electronically or recorded in a logbook maintained at the Field Equipment Center (FEC) as per the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). The functional check and tape length verification should be performed according to the instructions included in SESDFORM-043, *Well Sounder Function Check and Verification,* which also includes the form for recording the required information.
- These devices should be decontaminated according to the procedures specified in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) prior to use at the next well.

3 Water Level and Depth Measurement Procedures

3.1 General

The measurement of the groundwater level in a well is frequently conducted in conjunction with ground water sampling to determine the "free" water surface. This potentiometric surface measurement can be used to establish ground water direction and gradients. Groundwater level and well depth measurements are needed to determine the volume of water or drawdown in the well casing for proper purging.

All groundwater level and well depth measurements should be made relative to an established reference point on the well casing and should be documented in the field records. This reference point is usually identified by the well installer using a permanent marker for PVC wells, or by notching the top of casing with a chisel for stainless steel wells. By convention, this marking is usually placed on the north side of the top of casing. If no mark is apparent, the person performing the measurements should take both water level and depth measurements from the north side of the top of casing and note this procedure in the field logbook.

To be useful for establishing groundwater gradient, the reference point should be tied in with the NGVD (National Geodetic Vertical Datum) or a local datum. For an isolated group of wells, it is acceptable to use an arbitrary datum common to all wells in that group, if necessary.

Water levels should be allowed to equilibrate prior to measurement after removing sealing caps. There are no set guidelines and appropriate equilibration times can range from minutes to hours depending on well recharge, local geology and topography, and project objectives.

3.2 Specific Groundwater Level Measurement Techniques

Measuring the depth to the free ground water surface can be accomplished by the following methods. Method accuracies are noted for each of the specific methods described below.

- Electronic Water Level Indicators – These types of instruments consist of a spool of dual conductor wire, a probe attached to the end and an indicator. When the probe comes in contact with the water, the circuit is closed and a meter light and/or audible buzzer attached to the spool will signal contact. Penlight or 9-volt batteries are normally used as a power source. Measurements should be made and recorded to the nearest 0.01 foot.

- Other Methods – There are other types of water level indicators and recorders available on the market, such as weighted steel tape, chalked tape, sliding float method, air line pressure method and automatic recording methods. These methods are primarily used for closed systems or permanent monitoring wells.

Acoustic water level indicators are also available which measure water levels based on the measured return of an emitted acoustical impulse. Accuracies for these methods vary and should be evaluated before selection. Any method not capable of providing measurements to within 0.1 foot should not be used.

3.3 Special Considerations for Water Level Measurements at Sites with Shallow Groundwater Gradient

Groundwater gradients at some sites can be very shallow and if gradient and groundwater flow pattern (gradient direction) determination are part of the project objectives, it is critical that groundwater level measurements obtained from wells are as accurate as possible. Special care should be taken to allow the water level to equilibrate after removing sealing caps and the same sounder should be used for all measurements, if possible. The sounding activity should be coordinated to allow all wells to be sounded within the minimum possible time. This is particularly important in areas with potential tidal influences.

3.4 Total Well Depth Measurement Techniques

The well sounder, weighted tape or electronic water level indicators can be used to determine the total well depth. This is accomplished by lowering the tape or cable until the weighted end is felt resting on the bottom of the well. Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the tape end is touching the bottom of the well and sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements. The operator may find it easier to allow the weight to touch bottom and then detect the 'tug' on the tape while lifting the weight off the well bottom. All total depth measurements must be made and recorded to the nearest 0.1 foot. As a cautionary note, when measuring well depths with the electronic water level indicators, the person performing the measurement must measure and add the length of the probe beneath the circuit closing electrodes to the depth measured to obtain the true depth. This is necessary because the tape distance markings are referenced to the electrodes, rather than the end of the probe. For electronic sounders maintained at the SESD FEC, the sounder reel will be marked with the appropriate additional length identified as the 'TD adder'.

3.5 Equipment Available

The following equipment is available for ground water level and total depth measurements:

- Weighted steel measuring tapes
- Electronic water level indicators

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Groundwater Sampling**

Effective Date: March 6, 2013	**Number:** SESDPROC-301-R3

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Revision History

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SESDPROC-301-R3, Groundwater Sampling, replaces SESDPROC-301-R2. **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Changed author from Donald Hunter to Jonathan Vail. Changed Enforcement and Investigations Branch Chief from Archie Lee to Danny France. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **Section 2.3**: Item 4 was revised to reflect practice of using individual single-use preservative vials instead of preservatives prepared by ASB.	**March 6, 2013**
SESDPROC-301-R2, Groundwater Sampling, replaces SESDPROC-301-R1.	**October 28, 2011**
SESDPROC-301-R1, Groundwater Sampling, replaces SESDPROC-301-R0.	**November 1, 2007**
SESDPROC-301-R0, Groundwater Sampling, Original Issue	**February 05, 2007**

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting groundwater samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling groundwater samples in the field. On the occasion that SESD field personnel determine that any of the procedures described are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a groundwater sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

Columbia Analytical Services, Lab Science News, Passive Diffusion Devices & Polyethylene Diffusion Bag (PDB) Samplers.

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Puls, Robert W., Don A. Clark, and Bert Bledsoe. 1992. Metals in Ground Water: Sampling Artifacts and Reproducibility. Hazardous Waste and Hazardous Materials 9(2), pp. 149-162.

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SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurement, SESDPROC-102, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Groundwater Level and Well Depth Measurement, SESDPROC-105, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDROC-202, Most Recent Version

SESD Operating Procedure for Pump Operation, SESDPROC-203, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

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US EPA. 1995. Ground Water Sampling - A Workshop Summary. Proceedings from the Dallas, Texas November 30 – December 2, 1993 Workshop. ORD, Robert S. Kerr Environmental Research Laboratory. EPA/600/R-94/205, January 1995.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting groundwater samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting groundwater samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.

- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and placed in the project files.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Groundwater samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas unpreserved samples have only a seven-day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the groundwater has a high amount of dissolved limestone, i.e., is highly calcareous, there will most likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment, such as:

- Bailers must be constructed of stainless steel or Teflon®
- RediFlo2® submersible pumps used for sampling should be equipped with Teflon® sample delivery tubing
- Peristaltic pump/vacuum jug assemblies should be outfitted with Teflon® tubing from the water column to the transfer cap, which should also be constructed of Teflon®

2.2 Special Precautions for Trace Contaminant Groundwater Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.

- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Clean plastic sheeting will be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Groundwater samples will typically be collected from the discharge line of a pump or from a bailer, either from the pour stream of an up-turned bailer or from the stream from a bottom-emptying device. Efforts should be made to reduce the flow from either the pump discharge line or the bailer during sample collection to minimize sample agitation.

2. During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container.

3. Place the sample into appropriate, labeled containers. Samples collected for VOC, acidity and alkalinity analysis must not have any headspace. All other sample containers must be filled with an allowance for ullage.

4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. If additional preservative is needed, it should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).

2.4 Quality Control

If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same water-bearing formation. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 Groundwater Sampling Methods – Purging

3.1 General

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of actual aquifer conditions. In order to determine when a well has been adequately purged, field investigators should monitor, at a minimum, the pH, specific conductance and turbidity of the groundwater removed during purging and, in the case of permanent monitoring wells, observe and record the volume of water removed.

There are several purging strategies that may be used, depending on specific conditions encountered for given well sampling situations. When a specific well is characterized, based on the field investigators experience and knowledge, as having fairly typical water levels, depths and purge volumes, as determined according to the procedures in Section 3.2.1, below, SESD will normally use the multiple volume purging procedures and equipment described in Sections 3.2.1 and 3.3 of this procedure for purging the well.

When the traditional multiple volume purge method is considered and it is determined that excessive quantities of IDW would be generated using this method, it may be appropriate, under very limited and specific circumstances, to use an alternate method that reduces the time and amount of purge water to be removed prior to sampling the well. The field project leader will select the alternate method only after careful consideration of the conditions presented by the well and the impact these conditions have on all aspects of the sampling event (time required to sample, quantities of IDW requiring management, etc.).

The alternate purge procedures or sampling strategies available are the "Tubing-in-Screened Interval" method and the MicroPurge or No-Purge methods. These are described and discussed in Sections 3.2.2 and 4.5 of this operating procedure, respectively.

3.2 Purging Methods and Strategies

3.2.1 Traditional Multiple Volume Purge

3.2.1.1 Purging and Purge Adequacy

3.2.1.1.1 Purge Volume Determination

Prior to initiating the purge, the amount of water standing in the water column (water inside the well riser and screen) should be determined, if possible. To do this, the diameter of the well should be determined and the water level and total depth of the well should be measured and recorded. Specific methodology for obtaining these measurements is found in SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105).

Once this information is obtained, the volume of water to be purged can be determined using one of several methods. One is the equation:

$V = 0.041\ d^2h$

Where: h = depth of water in feet
d = diameter of well in inches
V = volume of water in gallons

Alternatively, the volume of standing water in the well and the volume of three water columns may be determined using a casing volume per foot factor for the appropriate diameter well, similar to that in Table 3.2.1. The water level is subtracted from the total depth, providing the length of the water column. This length is multiplied by the appropriate factor in the Table 3.2.1, corresponding to either the single well volume or the triple well volume, to determine both the single well volume and triple well volumes, in gallons, for the well in question. Other acceptable methods include the use of nomographs or other equations or formulae.

TABLE 3.2.1: WELL CASING DIAMETER VOLUME FACTORS

Casing Diameter (inches)	Gallons/ft, One Water Column	Gallons/ft, Three Water Columns
1	0.04	0.12
2	0.16	0.48
3	0.37	1.11
4	0.65	1.98
5	1.02	3.06
6	1.47	4.41
7	1.99	5.97
8	2.61	7.83
9	3.30	9.90
10	4.08	12.24
11	4.93	14.79
12	5.87	17.61

With respect to volume, an adequate purge is normally achieved when three to five well volumes have been removed. The field notes should reflect the single well volume calculations or determinations, according to one of the above methods, and a reference to the appropriate

multiplication of that volume, i.e., a minimum three well volumes, clearly identified as a purge volume goal.

3.2.1.1.2 Chemical Parameter Stabilization Criteria

With respect to the ground water chemistry, an adequate purge is achieved when the pH and specific conductance of the ground water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Primary Drinking Water Standard of 5 NTUs). Although 10 NTUs is normally considered the minimum goal for most ground water sampling objectives, lower turbidity has been shown to be easily achievable in most situations and reasonable attempts should be made to achieve these lower levels. (Note: Because groundwater temperature is subject to rapid changes when collected for parameter measurement, its usefulness is subject to question for the purpose of determining parameter stability. As such, it has been removed from the list of parameters used for stability determination. Even though temperature is not used to determine stability during well purging, it is still advisable to record the sample temperature, along with the other groundwater chemistry parameters during well purging, as it may be needed to interpret other chemical parameter results in some situations.)

Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU) and specific conductance varies no more than approximately 5 percent. Other parameters, such as dissolved oxygen (DO), may also be used as a purge adequacy parameter. Normal goals for DO are 0.2 mg/L or 10% saturation, whichever is greater. DO measurements must be conducted using either a flow-through cell or an over-topping cell to minimize or reduce any oxygenation of the sample during measurement. Oxidation Reduction Potential (ORP) should not be used as a purge stabilization parameter but may be measured during purging to obtain the measurement of record for ORP for the sampling event.

There are no set criteria for establishing how many total sets of measurements are adequate to document stability of parameters. If the calculated purge volume is small, the measurements should be taken frequently enough to provide a sufficient number of measurements to evaluate stability. If the purge volume is large, measurements taken every 15 minutes, for example, may be sufficient. See the SESD Operating Procedures for Field pH Measurement (SESDPROC-100), Field Specific Conductance Measurement (SESDPROC-101), Field Temperature Measurement (SESDPROC-102), Field Turbidity Measurement (SESDPROC-103), Field Measurement of Dissolved Oxygen (SESDPROC-106) and Field Measurement of Oxidation-Reduction Potential (SESDPROC-113) for procedures for conducting these measurements.

If, after three well volumes have been removed, the chemical parameters have not stabilized according to the above criteria, additional well volumes (up to five well volumes), should be removed. If the parameters have not stabilized within five volumes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible, with respect to turbidity. The conditions of sampling should be noted in the field log.

3.2.1.1.3 Purge Adequacy Considerations

In some situations, even with slow purge rates, a well may be pumped or bailed dry (evacuated). In these situations, this generally constitutes an adequate purge and the well can be sampled following sufficient recovery (enough volume to allow filling of all sample containers). ***It is not necessary that the well be evacuated three times before it is sampled***. The pH, specific conductance, temperature, and turbidity should be measured and recorded, during collection of the sample from the recovered volume, as the measurements of record for the sampling event.

For wells with slow recovery, attempts should be made to avoid purging them to dryness. This can be accomplished, for example, by slowing the purge rate. As water enters a well that has been purged to dryness, it may cascade down the sand pack and/or the well screen, stripping volatile organic constituents that may be present and/or introducing soil fines into the water column.

It is particularly important that wells be sampled as soon as possible after purging. If adequate volume is available immediately upon completion of purging, the well must be sampled immediately. If not, sampling should occur as soon as adequate volume has recovered. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered. Wells of this type should, unless it is unavoidable, not be purged at the end of one day and sampled the following day.

3.2.2 "Tubing-in-Screened-Interval" Method

The "Tubing-in-Screen" method, sometimes referred to as the "Low Flow" method, is used primarily when calculated purge volumes for the traditional purging method are excessive and present issues related to timely completion of the project and/or management of investigation derived waste.

3.2.2.1 Purge Criteria

3.2.2.1.1 Placement of Pump Tubing or Intake

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well. By definition, this method cannot be applied for purging with a bailer.

3.2.2.1.2 Conditions of Pumping

Prior to initiation of pumping, a properly decontaminated well sounder should be lowered into the well being sampled to monitor the static water level prior to and during the purging process. Ideally, there should be only a slight and stable drawdown of the water column after pumping begins. If this condition cannot be met, then one of the other methods should be employed.

3.2.2.1.3 Stability of Chemical Parameters

As with the traditional purging method described in Section 3.2.1, it is important that all chemical parameters be stable as defined in Section 3.2.1.1 prior to sampling.

3.3 Equipment Considerations for Purging

Monitoring well purging is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. The equipment utilized by Branch personnel will usually consist of peristaltic pumps and variable speed electric submersible pumps, but may also include bladder pumps or inertial pumps. The pump of choice is usually a function of the well diameter, the depth to water, the depth of the well and the amount of water that is to be removed during purging. Whenever the head difference between the sampling location and the water level is less than the limit of suction and the volume to be removed is reasonably small, a peristaltic pump should be used for purging. For wells where the water level is below the limit of suction (approximately 25’ to 30’, and/or where there is a large volume of water to be purged), the variable speed electric submersible pump would be the pump of choice. SESD Operating Procedure for Pump Operation (SESDPROC-203) contains the use and operating instructions for all pumps commonly used during SESD ground water investigations.

Bailers may also be used for purging in appropriate situations, however, their use is discouraged. Bailers tend to disturb any sediment that may be present in the well, creating or increasing sample turbidity. Bailers, if improperly used, may also strip volatile organic compounds from the water column being sampled. If a bailer is used, it should be a closed-top Teflon® bailer.

3.3.1 Wells Without Plumbing or In-Place Pumps

For permanent monitoring wells, the depth to water (water level) and depth of the well (total depth) should be determined before purging. Caution should be exercised during this procedure to prevent cross-contamination between wells. This is a critical concern when samples for trace organic compounds or metals analyses are collected. See SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) for cleaning procedures for well sounders. After cleaning, the well sounding device should be protected to keep it clean until its next use.

3.3.1.1 Purging with Pumps

3.3.1.1.1 Peristaltic Pumps

The following step-by-step procedures describe the process of purging with a peristaltic pump:

1. Cut a length of standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)) Teflon® tubing, equal to the well depth plus an additional five to ten feet. Enough tubing is needed to run from the ground surface up to the top of the well casing and back down to the bottom of the well. This will allow for operation of the pump at all possible water level conditions in the well.

2. Place one end of the tubing into the vacuum side of the peristaltic pump head. Proper sizing of the Teflon® and Silastic® or Tygon® tubing should allow for a snug fit of the Teflon® tubing inside the flexible tubing mounted in the pump head.

3. Run a short section of tubing (does not have to be Teflon®) from the discharge side of the pump head to a graduated bucket.

4. Place the free end of the Teflon® tubing into the well until the end of the tubing is just below the surface of the water column.

5. Secure the Teflon® tubing to the well casing or other secure object using electrician's tape or other suitable means. This will prevent the tubing from being lost in the well should the tubing detach from the pump head.

6. Turn on the pump to produce a vacuum on the well side of the pump head and begin the purge. Observe pump direction to ensure that a vacuum is being applied to the purge line. If the purge line is being pressurized, either switch the tubing at the pump head or reverse the polarity of the cables on the pump or on the battery.

7. If the pumping rate exceeds the recovery rate of the well, continue to lower the tubing into the well, as needed, until the drawdown stabilizes or the well is evacuated to dryness. If the pump is a variable speed peristaltic pump, and the water level in the well is being drawn down, reduce the speed of the pump in an attempt to stabilize the drawdown. If the well can be purged without evacuating the well to dryness, a sample with greater integrity can be obtained.

8. For wells which are not evacuated to dryness, particularly those with recovery rates equal to or very nearly equal to the purge rate, there may not be a complete exchange and removal of stagnant water in that portion of the water column above the tubing intake. For this reason, it is important that the tubing intake be placed in the very uppermost portion of the water column while purging. Standard field measurements should frequently be taken during this process to verify adequacy of the purge and readiness for sampling, as described in Section 3.

3.3.1.1.2 Submersible Pumps

When a submersible pump is used for well purging, the pump itself is lowered into the water column. The pump must be cleaned as specified in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

The pump/hose assembly used in purging should be lowered into the top of the standing water column and not deep into the column. This is done so that the purging will "pull" water from the formation into the screened area of the well and up through the casing so that the entire static volume can be removed. If the pump is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples, particularly if collected with a bailer, may not be representative of the aquifer conditions. It is recommended that the pump not be lowered more than three to five feet into the water column. If the recovery rate of the well is faster than the pump rate and no observable draw down occurs, the pump should be raised until the intake is within one foot of the top of the water column for the duration of purging. If the pump rate exceeds the recovery rate of the well, the pump will have to be lowered, as needed, to accommodate the drawdown. After the pump is removed from the well, the hose and the pump should be cleaned as outlined in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

3.3.1.2 Purging with Bailers

Standard-cleaned (SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206)), closed top Teflon® bailers with Teflon® coated stainless steel leaders and new nylon rope are lowered into the top of the water column, allowed to fill, and removed. It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during final stages of purging, to minimize turbidity and disturbance of volatile organic constituents. The use of bailers for purging and sampling is discouraged because the correct technique is highly operator dependent and improper use may result in an unrepresentative sample.

3.3.2 Wells With In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Many permanent monitoring wells at active facilities are also equipped with dedicated, in-place pumps. The objective of purging wells with in-place pumps is the same as with monitoring wells without in-place pumps, i.e., to ultimately collect a ground water sample representative of aquifer conditions. Among the types of wells identified in this section, two different approaches are necessary.

A permanent monitoring well with an in-place pump should, in all respects, be treated like a monitoring well without a pump. One limitation is that in most cases the in-place pump is “hard” mounted, that is, the pump is suspended in the well at a pre-selected depth and cannot be moved up or down during purging and sampling. In these cases, well volumes are calculated, parameters are measured and the well is sampled from the pump discharge, after volume removal and parameter conditions have been met.

In the case of the other types of wells, i.e., municipal, industrial and residential supply wells, however, not enough is generally known about the construction aspects of the wells to apply the same criteria as used for monitoring wells, i.e., 3 to 5 well volumes. The volume to be purged in these situations, therefore, depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following considerations and procedures should be followed when purging wells with in-place plumbing under the conditions described.

3.3.2.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be located between the

pump and the storage tank. If not, locate the valve closest to the tank. Measurements of pH, specific conductance, temperature, and turbidity are recorded at the time of sampling.

3.3.2.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Generally, under these conditions, 15 to 30 minutes will be adequate. Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

3.3.3 Temporary Monitoring Wells

3.3.3.1 General Considerations

Procedures used to purge temporary ground water monitoring wells differ from permanent wells because temporary wells are installed for immediate sample acquisition. Wells of this type may include standard well screen and riser placed in boreholes created by hand augering, power augering, or by drilling. They may also consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler or a Hydropunch® sampler. As such, the efforts to remove several volumes of water to replace stagnant water do not necessarily apply because stagnant water is not present. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply, to the extent possible, standard permanent monitoring well purging criteria to it to re-achieve aquifer conditions.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, resulting primarily in increased turbidity. Therefore, the goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low stress purging techniques using variable speed peristaltic pumps.

3.3.3.2 Purging When Water Level Is Within Limit of Suction

In situations where the elevation of the top of the water column is within the limit of suction (no greater than about 25 feet head difference between the pump and the water level), a variable speed peristaltic pump may be used to purge

temporary wells. Enough tubing is deployed to reach the bottom of the temporary well screen. At the onset of purging, the tubing is slowly lowered to the bottom of the screen and is used to remove any formation material which may have entered the well screen during installation. This is critical to ensuring rapid achievement of low turbidity conditions. After the formation material is removed from the bottom of the screen, the tubing is slowly raised through the water column to near the top of the column. The tubing can be held at this level to determine if the pump rate is drawing down the water level in the well. If the water level remains the same, secure the tubing at the surface to maintain this pumping level.

If drawdown is observed on initiation of pumping, reduce the pump speed and attempt to match the drawdown of the well. Sustained pumping at these slow rates will usually result in a relatively clear, low turbidity sample. If the drawdown stabilizes, maintain that level, however, if it continues to lower, "chase" the water column until the well is evacuated. In this case, the recovered water column may be relatively free of turbidity and can be sampled. It may take several episodes of recovery to provide enough volume for a complete sample.

3.3.3.3 Purging When Water Level Is Greater Than Limit of Suction

In situations where the elevation of the water table is greater than the limit of suction, peristaltic pumps cannot be used to purge temporary wells. If the temporary well is a ScreenPoint15® sampler with small diameter probe rod riser, the only practical choices for water removal are a small diameter bailer, a small diameter bladder pump or an inertial pump. If the well is to be used strictly for VOC screening, it may be acceptable to use the bailer to bail as much sediment from the well as possible prior to sampling. If metals are the analytes of concern, the bladder pump is the best choice for lowering the turbidity of the water column prior to sampling, followed next by the inertial pump. For larger diameter temporary wells, two-inch diameter or greater, bailers and the Grundfos® RediFlo2 may be used although excessive silt or other “fines” may present problems with the operation of the pump.

3.3.3.4 Considerations for Direct Push Groundwater Sampling

With many of the direct push sampling techniques, purging is either not practical or possible, therefore, no purging is conducted. The sampling device is simply pushed or driven to the desired depth and opened and the sample is collected and retrieved. As a result, some samples collected in this way may not be satisfactory or acceptable for certain analyses, i.e., the subject procedure may yield a turbid sample that is not appropriate for metals analyses.

3.4 Field Care of Purging Equipment

New plastic sheeting should be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they accidentally

come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool or contained in a large wash tub lined with plastic sheeting, both during transportation and during field use, to further minimize contamination by the transporting vehicle or the ground surface.

Careful consideration shall be given to using submersible pumps to purge wells which are excessively contaminated with oily compounds, because it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells of this type are encountered, alternative purging methods, such as bailers, should be considered.

3.5 Investigation Derived Waste

Purging generates quantities of purge water or investigation derived waste (IDW), the disposition of which must be considered. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

4 Groundwater Sampling Methods – Sampling

4.1 General

Sampling is the process of obtaining, containerizing, and preserving (if required) a ground water sample after the purging process is complete. Non-dedicated pumps for sample collection generally should not be used. Many pumps are made of materials such as brass, plastic, rubber, or other elastomeric products which may cause chemical interferences with the sample. Their principle of operation may also render them unacceptable as a sample collection device. It is recognized that there are situations, such as industrial or municipal supply wells or private residential wells, where a well may be equipped with a dedicated pump from which a sample would not normally be collected. Discretion should always be used in obtaining a sample.

4.2 Sampling Wells With In-Place Plumbing

Samples should be collected following purging from a valve or cold water tap as near to the well as possible, preferably prior to any storage/pressure tanks or physical/chemical treatment system that might be present. Remove any hose that may be present before sample collection and reduce the flow to a low level to minimize sample disturbance, particularly with respect to volatile organic constituents. Samples should be collected directly into the appropriate containers as specified in the ASBLOQAM. It may be necessary to use a secondary container, such as a clean 8 oz. or similar size sample jar or a stainless steel scoop, to obtain and transfer samples from spigots with low ground clearance. Also, refer to the discussion in the SESD Operating Procedure for Potable Water Supply Sampling (SESDPROC-305), Sec. 4.2, Potable Water Samples Collected from Wells with In-Place Plumbing**.** Potable well measurements for pH, specific conductance and turbidity and possibly temperature, if warranted, should be recorded at the time of sample collection.

4.3 Sampling Wells Without Plumbing, Within the Limit of Suction

4.3.1 Equipment Available

The pump of choice for sampling ground water within the limit of suction is the variable-speed peristaltic pump. Its use is described in the following sections. Other acceptable alternatives that may be used under these conditions are the RediFlo2® electric submersible pump (with Teflon® tubing) and a closed-top Teflon® bailer.

4.3.1.1 Peristaltic Pump, Direct from Pump Head Tubing

Samples for some constituents, primarily inorganic analytes such as metals and cyanide, may be collected directly from the pump head tubing. This method is acceptable under the following conditions:

- The pump head tubing must be changed between sampling locations;

- The pump head tubing must be either be certified clean according to SESD's internal quality control program described in Section 3.2 of the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) or
- An equipment rinsate blank is collected by pumping de-ionized water through a piece of the tubing.

4.3.1.2 Peristaltic Pump/Vacuum jug

It is not acceptable to collect samples for organic compound analyses through the flexible tubing used in the pump head. When collecting samples for organic compound analyses it is necessary to use a vacuum container, placed between the pump and the well for sample collection. The following step-by-step procedures describe the process of sampling with a peristaltic pump and vacuum jug (see note following these procedures for collection of VOC samples):

1. Disconnect the purge tubing from the pump. Make sure the tubing is securely attached to the protective casing or other secure object.

2. Insert the tubing into one of the ferrule nut fittings of a Teflon® vacuum container transfer cap assembly.

3. Place a suitable length of Teflon® tubing between the remaining transfer cap assembly ferrule nut fitting and the vacuum side of the flexible tubing in the peristaltic pump head. Securely hand-tighten both fittings.

4. Turn the pump on. Water should begin to collect in the transfer container (typically a 1-liter sample container) within a few minutes. If water does not begin to flow into the container within several minutes, check the transfer cap fittings and make sure the assembly is tightly attached to the container. It may be necessary to tighten the ferrule nuts with a wrench or pliers to achieve a vacuum in the system, particularly when approaching the maximum head difference between the pump and water table (limit of suction).

5. When the transfer container is nearly full, turn off the pump, remove the transfer cap assembly, and pour the sample into the appropriate containers. Because the 1-liter containers used by the Branch are rinsed with nitric acid during cleaning, they cannot be used for collecting samples to be analyzed for nitrogen sensitive parameters.

6. If additional sample volume is needed, replace the transfer cap assembly, turn the pump on, and collect additional volume. The use of Teflon® valves or ball check devices to retain the water column in the sample delivery tubing during the transfer phase, when large volumes of sample are required, is acceptable. These devices, however, must be constructed so that they may be completely disassembled and cleaned according to the procedures in SESD

Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205).

7. When sampling is completed, all Teflon® tubing should be discarded.

   NOTE: Samples for volatile organic compound analyses cannot be collected using this method. If samples for VOC analyses are required, they must be collected with a Teflon® or stainless steel bailer or by other approved methods, such as the "soda straw" method. The "soda straw" method involves allowing the tubing to fill, by either lowering it into the water column (A) or by filling it via suction applied by the pump head (B). If method (A) is used, the tubing is removed from the well after filling and the captured sample is allowed to drain into the sample vial. If method (B) is used, after running the pump and filling the tubing with sample, the pump speed is reduced and the direction reversed to push the sample out of the tubing into the vials. Avoid completely emptying the tubing when filling the sample vials when using method (B) to prevent introducing water that was in contact with the flexible pump head tubing. Either method is repeated, as necessary, until all vials are filled.

4.3.1.3 RediFlo2® Electric Submersible Pump (with Teflon® Tubing)

After purging has been accomplished with RediFlo2® electric submersible pump, the sample may be obtained directly from the pump discharge, provided that Teflon® tubing was used for the sample delivery line. The discharge rate of the pump should be reduced during volatile organic compound sample collection to minimize sample disturbance. Note, if the RediFlo2® electric submersible pump is used for sampling, the pump must undergo a full external and internal cleaning. In addition, pump rinsate blanks must be collected, at the appropriate frequency, to demonstrate that the pump has been adequately cleaned between wells.

4.3.1.4 Bailers

New bailer rope should be attached to the bailer via a Teflon® coated stainless steel wire. (If a bailer was used to purge the well, it may also be used to sample the well and new bailer rope is not required between purging and sampling). The bailer should be gently immersed in the top of the water column until just filled. At this point, the bailer should be slowly removed and the contents emptied into the appropriate sample containers.

4.4 Sampling Wells without Plumbing, Exceeding the Limit of Suction

All methods described previously in Section 4.3.2.1.3, RediFlo2® Electric Submersible Pumps, and Section 4.3.2.1.4, Bailers, are suitable sample methods where the water table is too deep to consider the use of a peristaltic pump for sampling.

4.5 Micro-Purge or No Purge Sampling Procedures

The Micro-Purge or No Purge sampling procedures are usually employed when it necessary to keep purge volumes to an absolute minimum. Among the Micro-Purge or No Purge procedures that might be employed are:

- Low pump rate sampling with peristaltic or submersible pumps (typical Micro-Purge sampling),
- HydraSleeve™ or
- Passive diffusion bag (PDB) sampling

The use of these procedures is acceptable only when the site hydrogeology is well understood, with respect to the hydraulic conductivity of geologic materials within the well screen interval. The underlying assumption, when employing these procedures, is that the formation in which the well is screened has a high hydraulic conductivity ($K>10^{-5}$ cm/sec, for example), resulting in a state of equilibrium existing between the water standing in the screened interval and the formation water in which the well is screened. In this situation, the well is considered to be in a perpetually "purged" state and purging is not required.

These procedures are generally impractical for SESD to implement because of the general lack of hydrogeologic information for the sampled wells and the real necessity, in some cases, that the pumps be pre-deployed to overcome issues related to turbidity resulting from pump placement prior to sampling.

4.5.1 Sampling with Pumps

The peristaltic pump tubing or intake point of the submersible pump is placed in the approximate mid-portion of the screened interval of the well or other interval selected by the field team leader. If turbidity and its impact on metals analyses are a concern, a period of time sufficient should be allowed to mitigate effects of pump or tubing placement. After it has been determined that sampling may proceed, the pump is turned on and operated at a rate that does not cause significant drawdown of the water column, as measured using a water level sounder. During sampling, sufficient water to supply enough volume for the analytes of concern and the purge parameters is pumped. Purging should continue until purge parameters stabilize, generally three consecutive stable sets of readings, before samples are collected.

4.5.2 HydraSleeves™

HydraSleeeves™ are grab sampling devices that are deployed in a closed configuration then opened in the desired interval for sample collection. The following is a summary of its operation:

1. Sampler placement - Reusable weight is attached and the HydraSleeve™ is lowered and placed at the desired position in the well screen. In-situ water pressure keeps the reed valve closed, preventing water from entering the sampler. Well is allowed to return to equilibrium.

2. Sample collection - The reed valve opens to allow filling when the sampler is moved upward faster than 1 foot per second, either in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval. There is no change in water level, and only minimal agitation during collection.

3. Sample retrieval - When the flexible sleeve is full, the reed valve closes and the sampler can be recovered without entry of extraneous overlying fluids. Samples are removed by puncturing the sleeve with the pointed discharge tube and draining the contents into containers for sampling or field measurement.

4.5.3 Passive Diffusion Bags

Passive diffusion bag (PDB) samplers are bags comprised of low-density polyethylene (LDPE) plastic and containing analyte-free water, preferably with no headspace. The bags are deployed, with stainless steel weights, to the desired sample interval and are allowed to equilibrate with the water at the point of deployment in the well. A deployment period of a minimum of 14 days is recommended to ensure equilibration prior to removal.

After 14 days, the bags and opened with a puncture device or other cutting implement and the contents transferred to containers for sampling or field measurement.

4.5.4 General Considerations for Micro-Purge or No-Purge Sampling

When using the Micro-Purge method, it may be advisable to deploy the tubing or pump in advance of sample collection. Introducing the tubing or pump into the screened interval is likely to dislodge sediment and other fines that have settled or bridged on the well screen material and the gravel pack media behind the screen. If sampling is conducted immediately, turbidity issues may render this method impractical from a parameter stability standpoint.

HydraSleevesTM and PDBs must be evaluated for appropriateness for analytes of concern.

4.6 Sample Preservation

After sample collection, all samples requiring preservation must be preserved as soon as practical. Consult the ASBLOQAM for the correct preservative for the particular analytes of interest. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

4.7 Special Sample Collection Procedures

4.7.1 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which comes into contact with the water in the well must be cleaned in accordance with the cleaning procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206). Pumps should not be used for sampling unless the interior and exterior portions of the pump and the discharge hoses are thoroughly cleaned. Blank samples should be collected to determine the adequacy of cleaning prior to collection of any sample using a pump other than a peristaltic pump.

4.7.2 Order of Sampling with Respect to Analytes

In many situations when sampling permanent or temporary monitoring wells, an adequate purge, with respect to turbidity, is often difficult to achieve. Removal and insertion of equipment after the purge and prior to actual sampling may negate the low turbidities achieved during purging and elevate turbidity back to unacceptable levels. For this reason, it is important that special efforts be used to minimize any disturbance of the water column after purging and to collect the aliquot for metals first. Therefore, the preferred order of sampling is metals first, followed by other inorganic analytes, extractable organic compounds and volatile organic compounds.

4.7.3 Filtering

As a standard practice, ground water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not allowed to correct for improperly designed or constructed monitoring wells, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis should not be filtered. Prior to filtration of the ground water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The monitoring wells, whether temporary or permanent, have been constructed and developed in accordance with the SESD Guidance Document, Design and Installation of Monitoring Wells (SESDGUID-001).

2. The ground water samples were collected using sampling techniques in accordance with this section, and the ground water samples were analyzed in accordance with USEPA approved methods.

3. Efforts have been undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the following:

   - Redevelopment or re-installation of permanent ground water monitoring wells.
   - Implementation of low flow/low stress purging and sampling techniques.

4. Turbidity measurements should be taken during purging and sampling to demonstrate stabilization or lack thereof. These measurements should be documented in the field notes. If the ground water sample appears to have either a chemically-induced elevated turbidity, such as would occur with precipitate formation, or a naturally elevated colloid or fine, particulate-related turbidity, filtration will not be allowed.

If filtration is necessary for purposes of geochemical modeling or other **pre-approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.

2. Use a 5 µm pore-size filter for the purpose of determining the colloidal constituent concentrations. A 0.1 µm pore-size filter should be used to remove most non-dissolved particles.

3. Rinse the cartridge or barrel-type filter with 500 milliliters of the solute (groundwater to be sampled) prior to collection of sample. If a membrane filter is used, rinse with 100 milliliters of solute prior to sample collection.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary. See Section 1.4, References, for complete citation for these documents.

Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes. See Section 1.4, References, for complete citation for these documents.

4.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

4.9 Auxiliary Data Collection

During ground water sample collection, it is important to record a variety of ground water related data. Included in the category of auxiliary data are water levels measured according to the SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105), well volume determinations (Section 3.1.1, Purging and Purge Adequacy), pumping rates during purging (see below), and occasionally, drillers or boring logs. This information should be documented in the field records.

4.9.1 Well Pumping Rate – Bucket/Stop Watch Method

The pumping rate for a pump can be determined by collecting the discharge from the pump in a bucket of known volume and timing how long it takes to fill the bucket. The pumping rate should be in gallons per minute. This method shall be used primarily with pumps with a constant pump rate, such as gasoline-powered or electric submersible pumps. Care should be taken when using this method with some battery-powered pumps. As the batteries' charge decreases, the pump rate also decreases so that pumping rate calculations using initial, high pump rates may be erroneously high. If this method is used with battery-powered pumps, the rate should be re-checked frequently to ensure accuracy of the pumping rate calculations.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **In Situ Water Quality Monitoring**

Effective Date: July 19, 2013	**Number:** SESDPROC-111-R3

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Revision History

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History	Effective Date
SESDPROC-111-R3, *In Situ Water Quality Monitoring*, replaces SESDPROC-111-R2 **General:** Corrected any typographical, grammatical, and/or editorial errors. **Cover Page:** Omitted John Deatrick as an author. Changed the Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. Changed the FQM from Laura Ackerman to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **Section 1.2:** Added the following statement – "Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use." **Section 1.4.2:** Replaced "instrument log book" with "Field Equipment Tracking System (FETS)."	July 19, 2013
SESDPROC-111-R2, *In Situ Water Quality Monitoring*, replaces SESDPROC-111-R1	December 7, 2009
SESDPROC-111-R1, *In Situ Water Quality Monitoring*, replaces SESDPROC-111-R0	November 1, 2007
SESDPROC-111-R0, In Situ Water Quality Monitoring, Original Issue	April 1, 2007

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Contents

1 General Information

1.1 Purpose

The purpose of this procedure is to document acceptable practices in the use of multiparameter data sondes in the monitoring of in situ water quality parameters and dye tracer.

1.2 Scope/Application

This procedure covers the use of multiparameter data sondes for monitoring of in situ water quality including real-time measurement, profiling, and unattended data logging.
In situ water quality parameters may include dissolved oxygen (DO), temperature, pH, conductivity, turbidity and chlorophyll. This procedure also applies to use of data sondes for monitoring dye tracer. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Precautions

1.4.1 Safety

Equipment must be handled in a safe manner. Safety issues related to calibration or measurement of a specific parameter are addressed in individual parameter procedures. In addition, safety precautions should be followed in the deployment of data sondes. For unattended deployment in wadeable systems, data sondes should only be deployed and retrieved under safe flow/stage conditions. When deploying from a bridge, an amber flashing light should be operated on the roof of the field vehicle. When deploying from a boat, standard boating safety procedures should be followed. The SESD Safety, Health and Environmental Management Program Procedures and Policy Manual, provides more information regarding field safety.

1.4.2 Equipment Handling

To ensure the safe and reliable operation of equipment, the manufacturers' directions for transport, cleaning, decontamination, storage and operation shall be followed. In general, upon return from the field and applicable data downloading, the batteries should be removed from the data sonde and the sonde washed via light brushing in warm, soapy water. Each probe should be cleaned and stored as directed by the manufacturer.

Prior to use, data sondes should be signed out in the Field Equipment Tracking System (FETS) according to SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). When unattended deployment is anticipated, pingers should be attached to the sonde, as feasible, to aid in recovery should the sonde be displaced during deployment.

1.4.3 Calibration

Prior to use, each sonde probe should be calibrated according to the specific parameter measurement procedure. However, because the sonde is a multi-probe unit, additional care must be taken to prevent cross-contamination of calibration standards. Similarly, calibration of multiple sonde units requires cross-contamination prevention procedures. Specifically, following immersion of the sonde probes into each calibration standard, all probes should be thoroughly rinsed in distilled or de-ionized water and the excess water shaken off or blotted dry with a lint-free wipe. Conductivity standards are much more sensitive to cross contamination/dilution than other standards; therefore, prior to immersion in a conductivity standard, all probes should be thoroughly rinsed and completely dried with lint-free wipes or compressed air. The conductivity probe on the sondes provides a linear reading of conductivity across the scale, so it is no longer necessary, as in some older technology meters, to calibrate with a standard close to what one may expect in the field. Therefore, due to the propensity of the standard to be easily diluted, one should use a relatively high concentration standard (typically in the 10,000 umho range) for conductivity calibrations.

Besides being easily diluted, conductivity also affects other parameters (specifically DO), therefore conductivity should always be the first parameter calibrated. The recommended order for calibration of the individual probes on a multiparameter sonde is as follows:

1. Conductivity
2. pH
3. DO
4. Turbidity/Chlorophyll/Rhodamine in any order

Rhodamine and Chlorophyll probes are calibrated in a similar fashion to turbidity. Specifically, the zero level is set using DI or distilled water followed by calibration to a known standard (typically 100 ppb for Rhodamine).

1.5 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Measurement of Dissolved Oxygen, SESDPROC-106, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

2 Methodology

2.1 General

With multiple probe options and customizable configuration, data sondes are extremely versatile tools for the measurement of in situ water quality. Effective use of multi-parameter sondes takes additional planning and procedures beyond those described in the individual operating procedures for each parameter (i.e., DO measurement, pH measurement, etc.).

Data sondes may be operated and/or programmed via the manufacturer's display unit or a laptop computer. In either case, it is recommended that the user take the manufacturer's applicable User Manual in the field should difficulties be encountered. If the display unit does not have a power indicator, the batteries should be checked or the unit charged, as applicable, prior to use. Power to the sonde may be supplied by the display unit or by the internal batteries installed in the sonde (a setting on the display unit menu). If the sonde is being powered by the display unit, it is possible to calibrate and set up the sonde for unattended deployment, when in fact there are no batteries in the sonde (the battery voltage being read is for the display unit and not for the sonde). Therefore, it is very important to insure that there are actually batteries in the sonde. Calibration and setting up for an unattended deployment use very little battery power, therefore, it is recommended that sondes be powered from their own internal batteries and not from the display unit. New alkaline or freshly charged nickel metal hydride (NMiH) batteries should be installed in each sonde prior to each field study. Generally, if the sondes will be deployed on multiple occasions during a field study, new alkaline batteries should be installed when the sonde voltage falls below 11.5 volts at end check. Nickel metal hydride (NMiH) batteries operate at a lower voltage than alkaline (1.2 volts vs. 1.5 volts); therefore, if using rechargeable batteries, they should be recharged or replaced if voltage falls below 10.5 volts.

Specific units require that, for the parameters of interest, the appropriate sensor be enabled via the display or laptop prior to use. The field investigator should follow manufacturer's procedures to ensure all required probes are functioning. If a particular parameter is not needed, the sensor should be turned off, via the menu, in order to conserve battery power. It should be noted that turning the reporting of the parameter off does not turn off the probe, it simply turns off the display of the parameter (the parameter is still being logged). One must go into the "Sensor" menu to actually turn off the sensor.

2.2 Real-time Monitoring

Real-time monitoring entails observing monitoring data via display unit or laptop computer as data is collected by the sonde. This data may be recorded in a field log book or logged to the internal memory of the sonde if so equipped. Logged data should be downloaded to a laptop or desktop computer as soon as possible. It is also recommended

that download files be backed up in a separate location (USB thumb drives work very well for this). In addition, even when logging data at regular intervals, it is recommend for real-time monitoring that data also be recorded in a field log book at some, likely less frequent, interval to ensure that some data is captured should the instrument logger fail.

Real-time monitoring generally involves hand-held deployment or attachment to a stationary object at the monitoring location. Hand-held deployments are useful for short-term monitoring in small, wadeable streams. For longer monitoring periods or to hold the sonde at a specific depth, attachment to a fixed object may be more effective. Fixed objects may include rocks or embedded logs already in place at the site or may include fence posts or rods placed by the field investigator prior to monitoring. Sondes may also be hung at desired depths from a boat on larger water bodies.

2.3 Profiling

Profiling involves real-time monitoring or individual measurements at several depths through a water column. Profiling is especially useful for documenting water column gradients or stratification of in situ parameters or for evaluating complete mix conditions in dye tracer studies. Profiling deployments are generally conducted by hand to provide the movement of the sonde through the water column; however, profiling can also be conducted using mechanical/ electrical wench or reel type devices. In profiling applications, the profiling cable should be labeled in some manner to indicate depth or the sonde calibrated for depth. In general, profiling data is recorded in a field log book along with the location and depth information for each measurement.

In fast moving waters it may be necessary to attach weight to the sonde. Weights should always be attached to the probe guard or sonde body, not the individual probes. If attached to the probe guard, weights should be secured in such a way that the weights and attachments do not interfere with probe operation. In all real-time and profiling applications, especially when the sonde is weighted, it is important to ensure that the profiling cable is securely attached to the baling harness of the sonde to prevent a disconnection of the sonde and potential loss or damage to the sonde.

It is important to note that SESD has two general type of sondes, vented and non-vented. Each type of sonde has its own profiling cable. The difference is how the depth sensor works. Non-vented sondes have a standard pressure or depth sensor that can be zeroed out at the site and will then accurately measure depth, typically to within a half a foot or less. Vented sondes have a small hole in the center of the connector pins where the cable attaches and are typically used to accurately (+/- 0.01 feet) measure changes in water stage level in unattended deployments, but may also be used for profiling applications. In order for a vented sonde to accurately measure depth or stage, the sonde MUST be used with a vented cable which vents to the atmosphere. If a vented sonde is used with a vented cable, just zero the depth at the site and measure depth as with a non-vented sonde If a vented sonde is used with a non-vented cable it will NOT give accurate depth

readings. Also, since vented sondes are typically used for stage measurements, the depth sensor is only rated to a maximum depth of 30 feet, whereas non-vented sondes are typically rated to 200 feet.

2.4 Unattended Deployment

Unattended deployment entails pre-programming and deployment of a sonde at a specific location to log monitoring data in the absence of observation by a field investigator. Unattended deployments are useful for collecting data at regular intervals over extended monitoring periods, frequently up to 3 – 4 days. However, since no data are recorded by hand during the deployment, it is critical that the sonde be correctly programmed.

Programming of the sonde should follow the manufacturer's procedures for unattended deployment. The sonde may be programmed in the lab prior to a field study or programmed in the field. Programming of the sonde is typically accomplished either by the sonde's display unit or by laptop computer. Programming requires input of a start data/time, deployment duration, data log file name, and monitoring interval. Programming times should always be input in local time for the study area, unless otherwise noted in the field log. The field log book should also include the sonde identifier, the date/time of initial deployment, date/time of retrieval, deployment location, and sonde depth. Similarly, recorded times should be in local time for the study area.

In addition to enabling the required probes as described in Section 2.1, some units further require identification of the parameters to include in the logged data file. The field investigator should follow manufacturer's procedures to ensure all necessary data will be successfully logged.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Logbooks**

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History	Effective Date
SESCPROC-010-R5, *Logbooks*, replaces SESDPROC-010-R4 **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Changed author from Liza Montalvo to Hunter Johnson. Changed Enforcement and Investigation Branch Chief from Archie Lee to Danny France. Changed Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. Changed Field Quality Manager from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history **Section 1.2**: Added the following statement: "Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002)." **Section 1.4**: Updated references **Section 2.1**: Added "bound" to the first sentence. Replaced the last sentence of the first paragraph with the following language: "All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information." Added the second paragraph partially comprised of language from the omitted paragraph from Section 2.2. **Section2.2**: Omitted the first paragraph. Added the following language to the first line (formerly the second paragraph): "The following requirements apply to **all logbooks**:" Added Items 1 and 2 and omitted Item 4. Renumbered items as appropriate. Added "End of Notes" to the second sentence in item 6 (formerly item 5). Also added the last sentence which states: "Field investigators will draw a diagonal line through blank or unused portions of pages/forms	May 30, 2013

that are located prior to the "End of Notes" entry and initial them." Added "and SESD-generated forms" to Item 7 (formerly item 6).	
SESCPROC-010-R4, *Logbooks*, replaces SESDPROC-010-R3	October 8, 2010
SESCPROC-010-R3, *Logbooks*, replaces SESDPROC-010-R2	November 1, 2007
SESCPROC-010-R2, *Logbooks*, Replaces SESDPROC-010-R1	September 25, 2007
SESDPROC-010-R1, *Logbooks*, Replaces *Field Records (Logbooks)* SESDPROC-010-R0	August 10, 2007
SESDPROC-010-R0, Field Records (Logbooks), Original Issue	February 5, 2007

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1 General Information

1.1 Purpose

This procedure is to be used by field investigators when documenting pertinent and factual information in logbooks related to field investigations involving sampling and measurement procedures and/or other data collection events.

1.2 Scope/Application

This document describes the various types of information that should be included in the field logbooks used to document field investigations conducted by SESD. Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002). Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

The procedures found within this document were prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Field Branches Quality Management Plan, SESDPLAN-001, Most Recent Version

2 Field Records and Documentation Procedures

2.1 General

Dedicated bound logbooks will be used for field data collection including but not limited to sampling, measurements and observations. Logbook entries should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information.

To facilitate accurate and complete documentation of field sampling and measurement activities, SESD-generated forms may be used. In order to be utilized, SESD-generated forms must be bound prior to use and adhere to all requirements outlined in this procedure. In cases where unbound pages/forms are necessary due to project requirements or practicality, approval must be obtained from both the Field Quality Manager and Management. In these cases, the Field Quality Manager and Management will dictate the steps that will be taken to ensure credibility, traceability and defensibility of information collected.

Any deviations from the quality assurance project plan that occur while in the field will be noted in the logbook(s). Logbook entries that may be considered privileged or confidential information will be handled in accordance with the relevant sections of SESD Operating Procedure for Control of Records (SESDPROC-002). The logbooks will be placed in the SESD project file upon transmittal of the final report to the project requestor.

2.2 Field Data Integrity and Accountability

The following requirements apply to **all logbooks**:

1. The project's unique identifier (unique identification number(s)) will be included on each page.
2. Field personnel will date and number each page. Numbering will be conducted by utilizing a format that incorporates both the current page number and the total number of pages (e.g. "page x of y" or "x/y", where "x" is the current page number and "y" is the total number of pages).
3. Observations, data and calculations will be recorded at the time they are made.
4. Unless prohibited by environmental conditions, pens with permanent ink will be used to record all data. When environmental conditions do not make it feasible to use permanent ink, entries should be made using a non-smear lead pencil (e.g., 2H or 3H). Upon returning from the field, the project leader will photocopy the penciled section of the logbook and certify, in writing, that the photocopied record is a true copy of the original logbook entry. The photocopy will be included in the project file.

5. Entries will be legible and contain pertinent, accurate and inclusive documentation of project activities.
6. Upon completion of the field investigation, the end of project entries in the logbook and/or bound forms will be clearly indicated. This may be accomplished by noting "End" or "End of Notes" on the last page of notes and dating and initialing the notation. Field investigators will draw a diagonal line through blank or unused portions of pages/forms that are located prior to the "End of Notes" entry and initial them.
7. In order to demonstrate continuity of the project and to preclude questioning of the integrity of the data collection process, pages and SESD-generated forms should not be removed from bound logbooks under any circumstances.
8. Data or other information that has been entered incorrectly will be corrected by drawing a line through the incorrect entry and **initialing and dating** the lined-through entry. Under no circumstances should the incorrect material be erased, made illegible or obscured so that it cannot be read.
9. If pre-printed adhesive labels are used in logbooks or bound forms to facilitate organization of information entry, the field investigator who is responsible for taking notes will sign the label with the signature beginning on the label and ending on the page of the logbook such that the label cannot be removed without detection.

2.3 Logbook Entry Information

2.3.1 General Information Required in All Logbooks

The following information will be included either on the front cover or the first page of **all logbooks**:

1. Project name
2. Project location
3. Project identification number
4. Project leader (full name)
5. Sample team leader (full name) and initials
6. Sample team member(s) (full name) and initials

2.3.2 Information Required for Sample Collection

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **samples** are collected:

1. Applicable SESD Operating Procedures for field sampling
2. Date and time of collection
3. Station identification
4. Sample identification

5. Method of collection
6. Number and type of containers
7. Sample collection equipment
8. SESD equipment identification number, if applicable
9. Physical description of sample
10. Matrix sampled
11. Sample team member duties (calibration, collection, deployment, etc.)
12. Sample preservation (including ice), if applicable
13. Conditions that may adversely impact quality of samples, if applicable (rain, wind, smoke, dust, extreme temperature, etc.)
14. GPS coordinates (Non-logging GPS units), if applicable
15. Location of electronic data file backups, if applicable

16. Monitoring of condition of ice in coolers or sampler
17. Other pertinent information.

2.3.3 Information Required for Field Measurements

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **measurements** are conducted:

1. Applicable SESD Operating Procedures for field measurement
2. Date and time of measurement or deployment
3. Sample identification, if appropriate
4. Station identification
5. Sample measurement equipment
6. SESD sample measurement equipment identification number
7. Manufacturer name, lot number and expiration date of all buffers and standards*
8. Calibration information, including before and after calibration readings*
9. Meter end check information
10. Deployment depth and total depth, if applicable
11. Pinger identification number and frequency for deployed equipment, if applicable
12. Time of retrieval for deployed equipment, if applicable
13. Physical description of matrix
14. Sample team member duties (calibration, collection, deployment, etc.)
15. Measurement values for non-logging equipment
16. GPS coordinates (non-logging GPS units), if applicable
17. Location of electronic data file backups, if applicable
18. Ambient air temperature, where applicable
19. Conditions that may adversely impact quality of measurement (Ex. temperature extremes)
20. Maintenance performed, if applicable

21. Meter malfunctions, if applicable
22. Other pertinent information

* Entry of calibration information in logbooks is only required for calibrations conducted in the field. All calibrations conducted at the Field Equipment Center or SESD laboratory will be recorded in the appropriate equipment tracking logbook.

2.3.4 Additional Information for Inclusion

The following information may be included in logbooks as appropriate:

1. Maps/sketches
2. Photographic or videographic log
3. Process diagrams

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Management of Investigation Derived Waste**

Effective Date: October 15, 2010	**Number:** SESDPROC-202-R2

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History	Effective Date
SESDPROC-202-R2, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R1. **General:** Corrected typographical, grammatical and/or editorial errors. **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo. **Revision History:** Changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement: Mention of trade names or commercial products does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted reference to the H: drive of the LAN. Changed Field Quality Manager to Document Control Coordinator. **Section 2, first bullet:** Replaced "personnel" protective equipment with "personal" protective equipment. **Table 1:** On the Hazardous/PPE-Reusable cell - Added the full reference to SESDPROC-205. On the Non-hazardous/Decontamination Water cell - Added a statement to reflect that decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plant representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations. On the Non-hazardous/Disposable Equipment cell - Added a statement to reflect that, if unfeasible, this equipment could be returned to the FEC for disposal in the FEC's dumpster.	October 15, 2010
SESDPROC-202-R1, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R0. **General** Corrected any typographical, grammatical and/or editorial errors. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch.	November 1, 2007

Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 1.5.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.	
SESDPROC-202-R0, Management of Investigation Derived Waste, Original Issue	February 05, 2007

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Table 1: Disposal of IDW **10**

Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures and considerations to be used and observed when managing investigation derived waste (IDW) generated during the course of hazardous waste site investigations.

1.2 Scope/Application

The procedures and management options for the different categories of IDW described in this document are to be used by SESD field personnel to manage IDW generated during site investigations. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to manage IDW generated at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when managing IDW. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when managing IDW:

- Due to time limitations and restrictions posed by RCRA regulations on storage of hazardous waste, accumulation start dates should be identified on all drums, buckets or other containers used to hold IDW so that it can be managed in a timely manner.
- During generation of both non-hazardous and hazardous IDW, keep hazardous IDW segregated from non-hazardous IDW to minimize the volume of hazardous IDW that must be properly managed.

2 Types of Investigation Derived Waste

Materials which may become IDW include, but are not limited to:

- Personal protective equipment (PPE) - This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.
- Disposable equipment and items - This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers (COLIWASAs), Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.
- Soil cuttings from drilling or hand augering.
- Drilling mud or water used for mud or water rotary drilling.
- Groundwater obtained through well development or well purging.
- Cleaning fluids such as spent solvents and wash water.
- Packing and shipping materials.

Table 1, found at the end of this procedure, lists the types of IDW commonly generated during field investigations and the current disposal practices for these materials.

For the purpose of determining the ultimate disposition of IDW, it is typically distinguished as being either hazardous or non-hazardous. This determination is based on either clear regulatory guidance or by subsequent analysis. This determination and subsequent management is the responsibility of the program site manager.

3 Management of Non-Hazardous IDW

Disposal of non-hazardous IDW should be addressed in the study plan or QAPP for the investigation. To reduce the volume of any IDW transported back to the Field Equipment Center (FEC), it may be necessary to compact the waste into a reusable container, such as a 55-gallon drum.

If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE, disposable equipment, and/or paper/cardboard into the facility's dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a nearby permitted landfill. On larger studies, waste hauling services may be obtained and a dumpster located at the study site.

Disposal of non-hazardous IDW such as drill cuttings, drilling mud, purge or development water, decontamination wash water, etc., should be specified in the approved study plan or QAPP. It is recommended that these materials be placed into a unit with an environmental permit, such as a landfill or sanitary sewer. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place these types of IDW into the facility's treatment system. It may be feasible to spread drill cuttings around the borehole, or, if the well is temporary, to place the cuttings back into the borehole. Non-hazardous monitoring well purge or development water may also be poured onto the ground down gradient of the monitoring well when site conditions permit. Purge water from private potable wells which are in service may be discharged directly onto the ground surface.

The minimum requirements for this subsection are:

- Non-hazardous liquid and soil/sediment IDW may be placed on the ground or returned to the source if doing so does not endanger human health or the environment or violate federal or state regulations. Under no circumstances, however, should monitoring well purge water be placed back into the well from which it came.
- Soap and water decontamination fluids and rinsates of such cannot be placed in any water bodies and must be collected and returned to the FEC for disposition.
- The collection, handling and proposed disposal method must be specified in the approved study plan or QAPP.

4 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW must be specified in the approved study plan or QAPP for the study or investigation. Hazardous IDW must be disposed as specified in USEPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed in the source area from which they originated if doing so does not endanger human health or the environment.

If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to allow arrangements for proper containerization, labeling, transportation and disposal/treatment in accordance with USEPA regulations.

The generation of hazardous IDW should be minimized to conserve Division resources. Most routine studies should not produce any hazardous IDW, with the possible exception of spent solvents and, possibly, purged groundwater. The use of solvents during field cleaning of equipment should be minimized by using solvent-free cleaning procedures for routine cleaning and decontamination (see SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205). If solvents are needed, the volume should be minimized by using only the amount necessary and by capturing the residual solvent separately from the aqueous decontamination fluids (detergent/wash water mixes and water rinses).

At a minimum, the requirements of the management of hazardous IDW are as follows:

- Spent solvents must be left on-site with the permission of site operator and proper disposal arranged.
- All hazardous IDW must be containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

Table 1: Disposal of IDW

TYPE	HAZARDOUS	NON - HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.
PPE-Reusable	Decontaminate as per SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, if possible. If the equipment cannot be decontaminated, containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Decontaminate as per SESDPROC-205, and return to FEC.
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator and arrange for proper disposal.	N/A
Soil Cuttings	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Groundwater	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Decontamination Water	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. Decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plant representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations.
Disposable Equipment	Containerize in DOT-approved container or 5-gallon plastic bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. If unfeasible, return to FEC for disposal in dumpster.
Trash	N/A	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.

**** These materials may be placed on the ground if doing so does not endanger human health or the environment or violate federal or state regulations.**

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Sample and Evidence Management**

Effective Date: January 29, 2013	**Number:** SESDPROC-005-R2

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SESDPROC-005-R2, *Sample and Evidence Management*, replaces SESDPROC-005-R1 **General:** Corrected any typographical, grammatical, and/or editorial errors. **General:** Replaced all references to FORMS with the generic term sample custody software program. The specific sections are listed below: Section 3.3 Section 3.4 Section 4.2 Section 5 **Title Page:** Changed the EIB Branch Chief from Antonio Quinones to Danny France. Changed the EAB Branch Chief from Bill Cosgrove to John Deatrick (Acting). Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis. **Revision History:** In the last sentence, changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement - Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance. Deleted reference to the H: drive. Section 2.2 Changed requirement for comments on sample label to make comments optional. Section 2.2.2 Removed requirement for printed copies of photographs in the official file. Section 3.3 Removed different custody requirements for criminal investigations so that all projects are treated consistently. Section 3.4 Removed statement in the fourth paragraph regarding retention of paper air bills in the official project file to reduce unnecessary paperwork. The air bill number or shipment tracking number is recorded on the chain of custody.	January 29, 2013

SESDPROC-005-R1, *Sample and Evidence Management*, replaces SESDPROC-005-R0 **General** Updated referenced procedures to reflect most recent version. Replaced "shall" with "will". **Cover Page:** Changed title for Antonio Quinones from Environmental Investigation Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Added reference for the SESD Operating Procedure for Control of Records. Alphabetized and revised the referencing style for consistency. **Section 2.2.3** Added that Confidential Business Information will be handled in accordance with SESD Operating Procedure for Control of Records.	November 1, 2007
SESDPROC-005-R0, Sample and Evidence Management, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when handling and managing samples and other types of evidence after their collection and during delivery to the laboratory.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when handling and managing samples and other evidence collected to support SESD field investigations. On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Digital Camera Guidance for EPA Civil Inspections and Investigations, July 2006

2 Sample and Evidence Identification

2.1 Introduction

Sample identification, chain-of-custody records, receipt for sample records and other field records will be legibly recorded with waterproof, non-erasable ink, unless otherwise specified. If errors are made in any of these documents, corrections will be made by crossing a single line through the error and entering the correct information. All corrections must be initialed and dated. If possible, all corrections should be made by the individual making the error.

Following are definitions of terms used in this section:

Field Investigator
Any individual who performs or conducts field sampling, observation and/or measurement activities in support of field investigations

Project Leader
The individual with overall responsibility for conducting a specific field investigation in accordance with this procedure

Field Sample Custodian
Individual responsible for identifying the sample containers and maintaining custody of the samples and the Chain-of-Custody Record

Sample Team Leader
An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team

Sampler
The individual responsible for the actual collection of a sample

Transferee
Any individual who receives custody of samples subsequent to release by the field sample custodian

Laboratory Sample Custodian
Individual responsible for accepting custody of samples from the field sample custodian or a transferee

One individual may fulfill more than one of the roles described above.

2.2 Sample and Evidence Identification Procedures

2.2.1 Sample Identification

The method of sample identification used depends on the type of sample collected. Field measurement samples are those collected for specific field analysis or measurement where the data are recorded directly in bound field logbooks or on the Chain-of-Custody Record. Examples of field measurements and analyses include XRF, pH, temperature, dissolved oxygen and conductivity. Samples collected for laboratory analysis will be identified by using a stick-on label or a tag which is attached to the sample container. In some cases such as biological samples, the label or tag may have to be affixed to a bag containing the sample. If a sample tag is used, the sample should be placed in a bag, then the sample and the tag will be placed in a second bag.

The following information will be included on the sample label or tag using waterproof, non-erasable ink:

- Project number;
- Field identification or sample station number;
- Date and time of sample collection;
- Designation of the sample as a grab or composite;
- Whether the sample is preserved or unpreserved;
- The general types of analyses to be performed.

Other information such as readily detectable or identifiable odor, color, or known toxic properties may be added as deemed necessary by the project leader or sample custodian.

2.2.2 Photograph, Digital Still Image and Video Identification

<u>Photographs and Digital Still Images</u>

When photographs or digital images are taken for purposes of documenting and supporting a field investigation, a record of each exposure or image will be kept in a bound field logbook. The following information will be recorded in the logbook:

- An accurate description of what the photograph or image shows, including orientation, if appropriate;

- The date and time that the photograph or image was taken;

- The name of the individual who took the photograph or digital image.

When photographs are taken with a film camera, the film should be developed with the negatives supplied uncut, if possible. The identifying information that was recorded in the field logbook will be entered on the back of the prints.

When digital images are obtained during a field investigation, an electronic copy of the unaltered investigation-related images will be placed in the official files. If deemed necessary due to project requirements, a printed copy of the original photographs may be placed in the official file. For enforcement cases, it is imperative that the individual who took the image be identified in the field logbook in the event their testimony is required.

Video

When a video recording is used as evidence in an enforcement case, the following information should be recorded in a bound field logbook:

- The date and time that the video was recorded;

- A brief description of the subject of the video;

- The person recording the video.

An audio record may also be included in the video tape with the above logistical information, as well as a narrated description of the video record.

A label will be placed on the video media with the appropriate identifying information (i.e., project name, project number, date, location etc.). In the event testimony regarding a video recording is required for an enforcement case, one individual should be responsible for recording the video for each case. The original, unaltered recording will be placed in the official files.

2.2.3 Identification of Physical Evidence

Physical evidence, other than samples, will be identified, when possible, by recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), the vessel or container should be marked with the field identification or sample station number for future identification. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking; however, these markings will be

recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain copies of recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. A unique identifier will be recorded on the document with that information as well as the following recorded in the logbook:

- Starting and ending time(s) and date(s) for the chart;
- An instantaneous measurement of the media being measured by the recorder will be taken and entered at the appropriate location on the chart along with the date and time of the measurement; and
- A description of the location being monitored and other information required to interpret the data such as type of flow device, chart units, factors, etc.

The field investigator will indicate who the chart (or copy of the chart) was received from and enter the date and time, as well as the field investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages and from whom they were received. Documents that are claimed by a facility to be "confidential" and, therefore, potentially subject to the Confidential Business Information requirements, will be handled in accordance with SESD Operating Procedure for Control of Records (SESDPROC-002).

3 Chain-of-Custody Procedures

3.1 Introduction

Chain-of-custody procedures are comprised of the following elements: 1) maintaining custody of samples or other evidence, and 2) documentation of the chain-of-custody for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample, or other evidence, from the moment of collection to its introduction into evidence.

3.2 Sample Custody

A sample or other physical evidence is in custody if:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or
- It is placed in a designated secure area.

3.3 Documentation of Chain-of-Custody

The following are used to identify and demonstrate how sample integrity is maintained and custody is ensured.

Sample Identification
A stick-on sample label or a tag should be completed for each sample container using waterproof, non-erasable ink as specified in Section 2.2.1.

Sample Seals
If appropriate, samples should be sealed as soon as possible following collection using a custody seal with EPA identification. The sample custodian or project leader will write the date and their initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 3.2, from the time of collection until the samples are delivered to the laboratory analyzing the samples.

Field Sample Custodian
The field sample custodian is the person designated by the project leader to receive and manage custody of samples while in the field, including labeling and custody sealing.

Chain-of-Custody Record

The field Chain-Of-Custody record is used to document the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or samples will be accompanied by a Chain-Of-Custody Record. This form may be generated by sample custody management software (Section 5) or it may be a pre-printed multi-sheet carbonless form for hand entry of required information. The Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory or other organizational elements. The Chain-of-Custody Record will not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 4, Receipt for Samples Form (CERCLA/RCRA/TSCA)). The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each final destination or laboratory used during the investigation.

All information necessary to fully and completely document the sample collection and required analyses must be recorded in the appropriate spaces to complete the field Chain-Of-Custody Record. The following requirements apply to Chain-Of-Custody records generated by either sample custody management software or by hand entry on pre-printed forms:

- All sampling team leaders must sign in the designated signature block.
- One sample should be entered on each line and not be split among multiple lines.
- If multiple sampling teams are collecting samples, the sampling team leader's name should be clearly indicated for each sample.
- The total number of sample containers for each sample must be listed in the appropriate column. Required analyses should be entered in the appropriate location on the Chain-of-Custody Record.
- The field sample custodian, project leader or other designee, and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurs should be documented in the proper space on the Chain-of-Custody Record. The exception to this requirement would be when packaged samples are shipped with a common carrier. Even though the common carrier accepts the samples for shipment, they do not sign the Chain-of-Custody Record as having received the samples.
- The last person receiving the samples or evidence will be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a uniquely identified document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope will be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for the envelope. Any time the EPA seal is broken, that fact will be noted on the Chain-Of-Custody Record and a new seal affixed, as previously described in this section.

Physical evidence such as video tapes or other small items will be placed in an evidence bag or envelope and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for these items. Any time the EPA seal is broken, that fact will be noted on the Chain-of-Custody Record and a new seal affixed.

EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented and the sample chain-of-custody can be established. If such samples are accepted, a standard sample label containing all relevant information and the Chain-Of-Custody Record will be completed for each set of samples.

3.4 Transfer of Custody with Shipment

Transfer of custody is accomplished by the following:

- Samples will be properly packaged for shipment in accordance with the procedures outlined in SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples (SESDPROC-209).

- All samples will be accompanied by the laboratory copy of the Chain-Of-Custody Record. If pre-printed forms are used, the white and pink sheets will be sent. If sample custody management software is used to generate the Chain-Of-Custody Record, the laboratory copy is identified with an “L” in the upper right corner. If multiple coolers are needed for shipment to a particular laboratory, the laboratory copy of the Chain-Of-Custody Record for the entire shipment is placed in a sealed plastic bag in one of the coolers. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-

Custody Record. One copy of the Record will be provided to and retained by the project leader. After samples have been received and accepted by the laboratory, a copy of the Chain-of-Custody Record, with ASB sample identification numbers, will be transmitted to the project leader. This copy will become a part of the project file.

- If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, an Air Bill should be used. The Air Bill number, shipment tracking number or registered mail serial number will be recorded in the remarks section of the Chain-Of-Custody Record.

4 Receipt for Samples Form (CERCLA/RCRA/TSCA)

4.1 Introduction

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions. The laws do not require that homeowners or other off-site property owners be given this form.

4.2 Receipt for Samples Form

If necessary, a Receipt for Samples form, using either the pre-printed form or one generated by sample custody management software, is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated (if a sample is split with a facility, state regulatory agency or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample). All information must be supplied in the indicated spaces to complete the Receipt for Samples form.

- The sampler(s) must sign the form in the indicated location

- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), a brief description of each sampling location and the total number of sample containers for each sample must be entered.

- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether split samples were received or declined and sign the form. The operator must give their title, telephone number and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.

The Receipt for Samples form is an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original form must be maintained in the project files.

5 Sample Custody Management Software

The container labels and the Chain-of-Custody record should be generated using a sample custody management software to streamline the documentation required by SESD and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. When possible, the sample custody management software should be used during all field investigations. Once the appropriate information is entered into the computer, the software will generate stick-on labels for the sample containers and will generate sample receipt forms and chain-of-custody records for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent for the bottle labels, sample receipt forms and chain-of-custody records. .

SOUTH CAROLINA DEPARTMENT OF
HEALTH AND ENVIRONMENTAL CONTROL

D H E C

PROMOTE PROTECT PROSPER

R.61-71
South Carolina Well Standards

Effective Date: April 26, 2002

Bureau of Water
2600 Bull Street
Columbia, S.C. 29201

DISCLAIMER

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SOUTH CAROLINA WELL STANDARDS
REGULATION 61-71

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(Statutory Authority: 1976 Code Section 44-55-40)

A. PURPOSE AND SCOPE.

These regulations establish minimum standards for the construction, maintenance, and operation of the following wells: individual residential, irrigation, monitoring (including non-standard installations), and boreholes to ensure that underground sources of drinking water are not contaminated and public health is protected. These regulations do not apply to public water wells as those standards are stated in R.61-58. Additional requirements may be found for injection wells in R.61-87; for water wells which produce greater than 3 million gallons per month in capacity use areas as stated in the regulations promulgated under the Groundwater Use and Reporting Act; for oil and gas exploration and production wells as required under the Oil & Gas Exploration, Drilling, Transportation and Production Act as stated in regulation R.121-8; and for monitoring and remediation wells required under the State Hazardous Waste Management Regulations (R.61-79). Permitting requirements for Individual Residential Wells and Irrigation Wells are found in R.61-44.

B. DEFINITIONS.

For the purpose of this regulation, the following terms are defined:

1. Annular Space - the space between the drill string or casing and the wall of the borehole.
2. Bored Well - a large diameter individual residential well or irrigation well, commonly equal to or greater than 24 inches in diameter, that is typically installed at a shallow depth and with casing constructed of rock, concrete, or ceramic material.
3. Borehole - a bored, drilled or driven shaft, or a dug hole, whose depth is greater than the largest surface dimension.
4. Borehole Completion - the date the total depth of the borehole has been reached.
5. Boring – a borehole for the purpose of sampling sub-surface materials such as environmental soil sampling borings, geotechnical borings, or exploration borings, but does not include boreholes completed as wells.
6. Casing - a pipe or tubing of appropriate material, of varying diameter and weight, lowered into a borehole during or after drilling in order to support the sides of the hole and thus prevent the sides of the hole from caving, to prevent loss of drilling mud into permeable strata, or to prevent fluids from entering or leaving the borehole.
7. Certified Well Driller - a driller duly and currently registered in South Carolina by the Department of Labor, Licensing, and Regulation.
8. Confining Layer - a geologic formation, group of formations, or part of a formation that is capable of significantly limiting fluid movement between hydrogeologic units.
9. Consolidated Formation or Material – crystalline, metamorphic, limestone, or otherwise competent rock.

10. Conventionally Installed Monitoring Well – a monitoring well where an annular space is created during the well construction process.

11. Development - the removal of formation cuttings, fine-grained sediments, drilling fluids, or additives from the borehole.

12. Direct Push Monitoring Well - a type of monitoring well constructed by pushing casing or other sampling device into the subsurface to obtain water samples for groundwater quality analysis, or to measure groundwater levels, where little or no annular space is created.

13. Environmental Soil Sampling Boring - a borehole used to obtain a soil sample for contamination investigations.

14. Exploration Boring - any borehole for the purpose of sub-surface mineral investigation and exploration.

15. Forced Injection of Grout - the emplacing of grout through a tremie pipe by pumping as opposed to pouring by gravity.

16. Filter Pack - an artificial filter material that is placed in the annular space around the well screen.

17. Geotechnical Boring – a typically shallow borehole for determining physical properties of the soil and subsurface, including foundation or general geotechnical borings and other such shallow borings incidental to construction activities.

18. Geothermal System Well – a type of well that is used to provide heat exchange for heating and cooling systems where the piping is below ground; includes, but is not limited to, closed loop and open loop systems.

19. Individual Residential Well - a well intended to produce potable water for human consumption at a single residence or a family.

20. Irrigation Well - a well intended to produce water for uses other than human consumption, to include, but not be limited to, lawn and landscape watering and agricultural uses.

21. Monitoring Well - any well constructed specifically to obtain a sample of groundwater for analysis, or any well used to measure groundwater levels. These wells include, but are not limited to, wells constructed using conventional drilling techniques and direct push methods.

22. Permanent Monitoring Well – any monitoring well that is intended for multiple sampling events over time.

23. Sanitary Cover - a removable seal to prevent the entrance of contaminants or foreign matter into the well.

24. Sanitary Seal - a removable seal at the top of the casing, between the casing and pipe, wire, tool, or device, capable of supporting such tools or devices, having no opening that would enable the entrance of contaminants or foreign matter into the well.

25. Screen – a filtering device that serves as the intake portion of a well that allows water to enter the well while preventing sediment from entering the well.

26. Surface Water – all water, which is open to the atmosphere and is subject to surface runoff that includes lakes, streams, ponds, ditches, and reservoirs.

27. Temporary Monitoring Well – a monitoring well where a one-time groundwater sample or groundwater level measurement is obtained.

28. Thermoplastic - materials composed of acrylonitrile-butadiene-styrene (ABS), polyvinyl chloride (PVC), or styrene-rubber (SR) plastics.

29. Tremie - the use of a small diameter pipe inserted into the borehole through which the filter pack or grout is placed at the desired depth to either complete construction of the well or to abandon the boring.

30. Unconsolidated Formation or Material - layers or sequences of sands, silts, or clays.

31. Vent – a device to keep foreign matter out of the well and that allows the well to be vented to the atmosphere.

32. Well – any borehole completed for the purpose of extracting or injecting fluid. This shall include, but not be limited to, wells used for irrigation, individual residential drinking water, environmental restoration, geothermal well systems, or environmental sampling. Wells fall into one of the following types:

    a. Type I - open hole in bedrock aquifers;

    b. Type II - screened with natural filter in unconsolidated aquifers;

    c. Type III - screened with artificial filter in unconsolidated aquifers;

    d. Type IV - open hole in consolidated limestone aquifers;

    e. Type V - bored or dug well having a large diameter.

33. Well Completion Date – the date the casing has been grouted.

C. VARIANCES.

Any requests for variances to these regulations shall be directed in writing to the Department and shall be considered on a well-specific basis by the Department. A variance can be issued as an alternative construction method that ensures the equivalent protection of the groundwater resource and public health when the standards in this regulation cannot otherwise be met. The Department may revoke issued variances as determined to be appropriate by the Department.

D. GENERAL.

1. All wells shall be drilled, constructed, and abandoned by a South Carolina certified well driller per 40-23-10 et seq.

2. Analytical data submitted to the Department shall be from a South Carolina Certified Laboratory per R.61-81.

E. LOCATION OF WELLS.

1. Wells outlined in Section F and G shall be located the specified minimum distance from all of the following existing potential sources of contamination:

   a. Sewer lines .....20 feet

   b. Lakes, streams, surface-water bodies.....50 feet

   c. Septic tank/tile fields.....50 feet

   d. Animal feedlots, barns, stables.....50 feet

   e. Waste disposal--land application sites ..... 100 feet

   f. Waste treatment lagoons ..... 100 feet

   g. Chemical, herbicide, pesticide and petroleum storage or handling sites..... 100 feet

   h. Landfills ..... 100 feet

   i. Hazardous waste landfills/surface impoundments ..... 100 feet

   j. Radioactive waste landfills ..... 100 feet

   k. Property lines and buildings.....5 feet

2. Certain conditions may require increased distances of certain wells from potential contamination sources or known contamination and the decision shall be made in consultation with the Department. These include but are not limited to:

   a. Type I and IV wells with less than twenty feet of casing

   b. Type I wells where fractured rock is at the surface

   c. Type II or III wells with no confining layers between the screened zone(s) and the ground surface

   d. Type IV wells where no confining layers overlie the open-hole limestone or the limestone is at shallow depth

   e. Type V wells because they are under water-table conditions

3. At the time of construction, all wells shall be accessible for cleaning, treatment, repair, inspection, and other attention as may be necessary.

F. INDIVIDUAL RESIDENTIAL AND IRRIGATION WELLS.

1. Drilling.

   a. The drilling process or use of drilling fluid additives shall not contaminate any aquifer.

   b. Water used in the drilling process shall be obtained from a source that will not result in chemical or biological contamination of any aquifer. Water taken directly from ponds, lakes, streams or other surface water sources shall not be used.

2. Grouting.

a. The diameter of the drilled hole shall be large enough to allow for a minimum of 1.5 inches of annular space on all sides of the casing for forced injection of grout through a tremie pipe.

b. Grout is to be composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.

(1) Neat cement grout shall be composed of Class A, Type I Portland Cement mixed with not more than seven (7) gallons of clean water per bag (one cubic foot or 94 pounds) of cement with a density of 15 to 16 pounds per gallon, or to manufacturer's specifications.

(2) Bentonite-cement grout shall be composed of powdered bentonite (less than 5% by weight) mixed at not more than 8 gallons of water to the bag, with a density of 14 to 15 pounds per gallon, or to manufacturer's specifications.

(3) High solids sodium bentonite grout shall have a minimum of 20% solids and be mixed per manufacturer's specifications with water and/or other required additives.

c. Grout shall fill the entire annular space from a minimum depth of twenty feet from the land surface at the time of well completion, unless otherwise approved by the Department. While the Department discourages wells less than 20 feet to be used for human consumption, it is recognized that due to differing hydrogeologic conditions across the state, some wells may need to be screened or completed at depths less than 20 feet to obtain potable water in the shallow aquifer. Therefore, wells less than 20 feet in depth, but no less than 15 feet in depth, are allowed. In these cases, in Type II and Type III wells, grout shall extend from one foot above the screen to the land surface and, in Type I and Type IV wells, the entire annular space shall be filled with grout. Any other minimum well depths may only be allowed per the variance procedure outlined in this regulation.

d. All grouting shall be accomplished using forced injection to emplace the grout. When emplacing the grouting material, the tremie pipe shall be lowered to the bottom of the zone to be grouted. The tremie pipe shall be kept full continuously from start to finish of the grouting procedure, with the discharge end of the tremie pipe being continuously submerged in the grout until the zone to be grouted is completely filled.

e. Wells shall be grouted in-place within five (5) days after borehole completion.

f. When high solids sodium bentonite grouts are used, a vapor barrier at the land surface at least the width of the annular space made of suitable materials, as approved by the Department, such as native soils, gravel,

sand, or thermoplastic material, is required for public safety and structural stability of the well.

3. Plumbness and Alignment.

Wells shall be constructed sufficiently plumb and straight so as to cause no interference with intended use.

4. Development.

a. Wells shall be properly developed. In a screened well, development shall be by a method that ensures that water is forced to flow into and out of the screen.

b. Development shall be complete when the well produces water typical of the aquifer being utilized.

5. Well Tested for Yield.

If a yield test is conducted, it shall be by a standard method and accurately measure flow. Results are to be included with the well record to be sent to the Department.

6. Backflow Prevention.

Approved backflow prevention devices are required on all wells that utilize a chemical feed system for any purpose other than water treatment. The backflow prevention device shall be installed so as to preclude any direct pathway for any contaminant to enter an underground source of drinking water.

7. Disinfection.

All individual residential wells and irrigation wells shall be disinfected upon well completion. The well shall also be disinfected upon any well maintenance, repair, pump repair, pump installation, or testing. Disinfectants shall be placed in the well in order to provide a chlorine residual from 50 ppm (milligrams per liter) to 250 ppm for a minimum of four hours before being flushed from the well. The method of chlorination shall be one that insures that the chlorine is uniformly distributed in the well. The well shall be flushed sufficiently after disinfection to remove the disinfectant and to condition the well for use.

8. Well Identification.

These wells shall be properly labeled with an identification plate immediately upon well completion. The identification plate shall be constructed of a durable, weatherproof, rustproof, material. The identification plate shall be permanently secured to the well casing or enclosure floor around the casing where it is readily visible. The identification plate shall be permanently marked to show:

a. Company name and certification number of the driller who installed the well;

b. Date well was completed;

c. Total depth (feet); and,

d. Casing depth (feet).

9. Materials.

a. Casing.

(1) Casing may be driven, lowered, or installed in any manner that will effect a continuous water tight and plumb installation.

(2) A well point, drive pipe, or drive shoe shall be structurally suitable to prevent rupture during the driving of the casing.

(3) Permanent well casing shall be new, seamless, or electric-resistance welded steel or galvanized pipe or thermoplastic pipe.

(4) Casing shall have watertight joints that shall be glued or threaded and coupled if plastic, or electrically welded or threaded and coupled with heavy recessed-type couplings of steel, if metal.

(5) Casing shall be of standards classified by the American Society for Testing and Materials (ASTM) and shall be NSF approved.

(6) New steel casing which bears mill markings and which conforms to the standard and specifications ASTM A-53, ASTM A-120, or American Petroleum Institute (API-5L) for water well pipe shall be used.

(7) Casing weighing less than "standard weight" steel pipe is allowed by the Department for water wells when the following requirements are met:

(a) The casing shall have a minimum nominal wall thickness of 0.188 inches;

(b) Casing meets the requirements of American National Standards Institute and ASTM (ANSI/ASTM) for water well casing.

(8) In consolidated formations, the well casing shall be set into the formation so as to provide a watertight seal between the casing and the top of the consolidated formation.

(9) In unconsolidated material, well casing shall be set into the first confining layer or to twenty feet, whichever is greater.

(10) Casing installed shall extend a minimum of one foot above the land surface at the time of construction. If an above ground pump is used, the casing may extend less than one foot above the land surface or below the land surface with a protective wellhead cover that allows access to the wellhead if the piping connecting the well casing to the pump is glued or threaded and is watertight.

(11) Thermoplastic casing may be used provided the casing:

(a) Conforms to requirements of American National Standards Institute/American Society of Testing and Materials (ANSI/ASTM), specification F480-77 for thermoplastic water well casing pipe and couplings made in standard dimension ratios (SDR);

(b) Has minimum wall thickness and tolerances to meet or exceed requirements for SDR 26 thermoplastic water well casing pipe and couplings made in standard dimension ratios;

(c) Has wall thickness and tolerances to meet or exceed requirements for Schedule 40 thermoplastic water well casing pipe for nominal sizes three inches or smaller;

(d) Can be installed without interference from formational material or other objects that may cause physical damage to the casing during emplacement.

b. Screens.

(1) The well, if constructed to obtain water from an unconsolidated formation, shall be equipped with a screen that will prevent the entrance of formation material into the well after the well has been developed and completed.

(2) The well screen shall meet standards classified by the American Society for Testing and Materials (ASTM) and shall be National Sanitation Foundation (NSF) approved material, and shall be of a strength to satisfactorily withstand chemical or physical forces applied to it during and after installation.

(3) Thermoplastic well screens shall:

(a) Have a minimum wall thickness and tolerance which meet or exceed requirements for schedule 40 thermoplastic water well casing pipe for nominal sizes three inches and smaller;

(b) Have a minimum wall thickness and tolerance which meet or exceed requirements for SDR 26 thermoplastic water well casing for nominal sizes greater than three inches, and;

(c) Be installed in wells without interference from formation material or other objects that may cause physical damage during emplacement and do not exceed manufacturers recommendations for depth placement.

(d) Multi-screened wells shall not connect aquifers or zones that have documented differences:

(1) In water quality that would result in contamination of any aquifer or zone such that any State Primary Drinking Water standard is exceeded;

(2) In static water levels that would result in depletion of water from any aquifer or zone, or significant loss of head in any aquifer or zone.

(e) The bottom of the lower-most well screen shall be plugged or capped.

10. Filter Pack.

When a filter pack is used, the following criteria shall apply:

a. The filter pack shall be composed of uniformly sized, quartz sand or gravel being free from clay, silt, or other deleterious material.

b. The filter pack shall be installed using a method that ensures placement into the annular space around the screens.

c. The filter pack shall be disinfected prior to or during installation.

d. The filter pack shall not connect aquifers or zones that have documented differences:

(1) In water quality that would result in contamination of any aquifer or zone such that any State Primary Drinking Water standard is exceeded;

(2) In static water levels that would result in depletion of water from any aquifer or zone, or significant loss of head in any aquifer or zone.

11. Sanitary Seal.

a. A sanitary seal shall be provided on the top of the well casing.

b. If a vent is used, it shall be of a type to prevent the entrance of contaminants, insects, or rainwater into the well.

12. Reporting.

a. The Water Well Record Form 1903, or other approved form, shall be completed and submitted to the Department by the contractor within thirty days after well completion. In addition to the water well record, the driller shall submit additional information as available such as chemical or bacterial results, if taken, and pumping information.

b. A Form 1903 shall also be submitted for individual residential and irrigation wells that are abandoned.

13. Operations and Maintenance.

    a. All wells shall be operated and maintained at all times in such a manner so as to protect underground sources of drinking water from contamination and to protect public health. The well owner may be required to provide additional security against vandalism as appropriate. The well owner is responsible for routine maintenance and operation of the well.

    b. The well driller is responsible for ensuring wells are constructed in accordance with this regulation. Once the well driller has provided a properly constructed well to the well owner, the well driller is not responsible for normal wear of the well.

    c. A sampling spigot shall be installed on the wellhead.

14. Abandonment.

    a. When any well is removed from service or prior to putting in service, the well shall be sealed with a watertight cap or seal. The well shall be maintained such that it is not a source or channel of contamination while it is not in service. Until a well is abandoned, all provisions for protection of the water against contamination and for maintaining sanitary conditions around the well shall be carried out to the same extent as though the well were in routine use. This goal shall be met when conducting repair or maintenance on the well, surrounding structures, or pumps.

    b. Any well removed from service for longer than thirty-six months shall be permanently abandoned unless a variance from the Department is requested.

    c. Any well that acts as a source of contamination shall be repaired or permanently abandoned immediately after receipt of notice from the Department.

    d. Abandonment shall be by forced injection of grout or pouring through a tremie pipe starting at the bottom of the well or fill material and proceeding to the surface in one continuous operation.

    e. When an individual residential well or irrigation well is permanently abandoned, at a minimum, the well may be filled with either bentonite-cement, neat cement, 20% high solids sodium bentonite grout, sand, or gravel to no closer than twenty feet below the ground surface. The remaining twenty feet to the ground surface shall be filled with neat cement, bentonite-cement, or 20% high solids sodium bentonite grout.

G. BORED INDIVIDUAL RESIDENTIAL AND IRRIGATION WELLS.

The construction of bored wells shall meet the following minimum requirements:

1. Drilling.

a. The drilling process or use of drilling fluid additives shall not contaminate any aquifer.

b. Water used in the drilling process shall be obtained from a source that will not result in chemical or biological contamination of any aquifer. Water taken directly from ponds, lakes, streams or other surface water sources shall not be used.

2. Grouting.

a. The diameter of the borehole shall be large enough to allow for a minimum of 1.5 inches of annular space on all sides of the casing for forced injection of grout through a tremie pipe.

b. Grout is to be composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.

(1) Neat cement grout shall be composed of Class A, Type I Portland Cement mixed with not more than seven (7) gallons of clean water per bag (one cubic foot or 94 pounds) of cement with a density of 15 to 16 pounds per gallon, or to manufacturers specifications

(2) Bentonite-cement grout shall be composed of powdered bentonite (less than 5% by weight) mixed at not more than 8 gallons of water to the bag, with a density of 14 to 15 pounds per gallon, or to manufacturers specifications.

(3) High solids sodium bentonite grout shall have a minimum of 20% solids and be mixed per manufacturers specifications with water and/or other required additives.

c. When high solids sodium bentonite grouts are used, a vapor barrier at the land surface made of suitable natural materials as approved by the Department, such as native soils, gravel, or sand, is required.

d. Grout shall fill the entire annular space from a minimum depth of fifteen feet from the land surface at the time of well completion to ensure that water does not enter the well from the joints in the well casing. Bored wells shall be greater than 15 feet in depth.

e. Grouting shall be done by forced injection of grout. When emplacing the grouting material, the tremie pipe shall be lowered to the bottom of the zone to be grouted. The tremie pipe shall be kept full continuously from start to finish of the grouting procedure, with the discharge end of the tremie pipe being continuously submerged in the grout until the zone to be grouted is completely filled.

f. Grouting shall take place within five (5) days after borehole completion.

3. Development.

Development shall be complete when the well produces water typical of the aquifer being utilized.

4. Well Tested for Yield.

If a yield test is conducted, it shall be by a standard method and accurately measure flow. Results are to be included in the well record to be sent to the Department.

5. Backflow Prevention.

Approved backflow prevention devices are required on all wells that utilize a chemical feed system for any purpose other than water treatment. The backflow prevention device shall be installed so as to preclude any direct pathway for any contaminant to enter an underground source of drinking water.

6. Disinfection.

All individual residential wells shall be disinfected upon the initial installation of the pump and sanitary cover. The well shall also be disinfected upon any subsequent well maintenance, repair, pump repair, pump installation, or testing. Disinfectants shall be placed in the well in order to provide a chlorine residual from 50 ppm (milligrams per liter) to 250 ppm for a minimum of four hours before being flushed from the well. The method of chlorination shall be one that insures that the chlorine is uniformly distributed in the well. The well shall be flushed sufficiently after disinfection to remove the disinfectant and to condition the well for use.

7. Well Identification.

All wells shall be properly labeled with an identification plate immediately upon well completion. The identification plate shall be constructed of a durable, weatherproof, rustproof, material. The identification plate shall be permanently secured to the well casing or enclosure floor around the casing where it is readily visible. The identification plate shall be permanently marked to show:

a. Company name and driller's certification number who installed the well;

b. Date well was completed;

c. Total depth (feet);

d. Casing depth (feet).

8. Casing.

a. Casing may be driven, lowered, or installed in any manner that will effect a continuous water tight and plumb installation.

b. Any piping connecting through the well casing shall be watertight.

9. Filter Pack.

When a filter pack is used, the following criteria shall apply:

a. The filter pack shall be composed of uniformly sized, quartz sand or gravel being free from clay, silt, or other deleterious material.

b. The filter pack shall be installed using a method approved by the Department into the annular space.

c. The filter shall be disinfected prior to or during installation.

10. Sanitary Cover.

A sanitary cover shall be provided on the top of the well casing.

11. Reporting.

a. The Water Well Record Form 1903, or other approved form, shall be completed and submitted to the Department by the contractor within thirty days after well completion. In addition to the water well record, the driller shall submit additional information as available such as chemical or bacterial results, if taken, and pumping information.

b. A Form 1903 shall also be submitted for bored wells that are abandoned.

12. Operations and Maintenance.

Bored wells shall be operated and maintained at all times in such a manner so as to protect underground sources of drinking water from contamination and to protect public health. The well owner may be required to provide additional security against vandalism as appropriate. The well driller is responsible for ensuring wells are constructed in accordance with this regulation. Once the well driller has provided a properly constructed well to the well owner, the well driller is not responsible for normal wear of the well. The well owner is responsible for maintenance and operation of the well.

13. Abandonment.

a. When a bored well is removed from service or prior to putting in service, the well shall be covered with a cap or seal. The well shall be maintained such that it is not a source or channel of contamination while not in service. Until a well is abandoned, all provisions for protection of the water against contamination and for maintaining sanitary conditions around the well shall be carried out to the same extent as though the well were in routine use. This goal shall be met when conducting repair or maintenance on the well, surrounding structures, or pumps.

b. Any well removed from service for longer than thirty-six months shall be permanently abandoned unless a variance from the Department is requested.

c. Any well that acts as a source of contamination shall be repaired or permanently abandoned immediately after receipt of notice from the Department.

d. Abandonment shall be by forced injection of grout or pouring through a tremie pipe starting at the bottom of the well or fill material and proceeding to the surface in one continuous operation, unless compacted clay is used.

e. The bored well shall be abandoned immediately upon being permanently taken out of service. The well may be filled with either bentonite-cement, neat cement, 20% high solids sodium bentonite grout, sand, or gravel to no closer than twenty feet below the ground surface. The remaining twenty feet to the ground surface shall be filled with neat cement, bentonite-cement, or 20% high solids sodium bentonite grout, or compacted clay.

H. MONITORING WELLS

1. Requirements For All Permanent and Temporary Monitoring Wells.

a. Due to the nature and purpose of a monitoring well, the depth and location requirements in respect to surface water bodies, potential contamination sources, etc., are variable, and shall be approved on a case-by-case basis by the Department. All monitoring wells shall have Department approval prior to installation or abandonment. Prior to the construction of any monitoring well, the following information shall be completed on a form provided and/or approved by the Department and shall be submitted to the Department:

(1) Proposed well location(s) on a scaled map or plat;

(2) Proposed well construction details;

(3) Intended purpose of the well(s);

(4) Well owner's name and mailing address;

(5) Property owner's name and mailing address, if different from the well owner;

(6) Mailing address and county of location where monitoring wells are to be installed, if different from the well owner's or property owner's address;

(7) Proposed parameters to be analyzed; and,

(8) Proposed drilling date.

If any of the information provided to the Department changes, the Department shall be notified at least 24 hours prior to well construction.

b. All monitoring wells shall be drilled, constructed, maintained, operated, and/or abandoned to ensure that underground sources of drinking water are not contaminated.

c. All monitoring wells shall yield water samples and water levels that are representative of the zone monitored.

d. The well owner shall submit all analytical data and water levels obtained from each monitoring well to the Department within 30 days of receipt of laboratory results unless another schedule has been approved by the Department.

e. Any monitoring well which is destroyed, rendered unusable, or abandoned, shall be reported to the Department, and shall be properly abandoned, revitalized, or replaced as appropriate or as required by permit or regulation.

f. A Water Well Record Form 1903 or other form provided and/or approved by the Department shall be completed and submitted to the Department within 30 days after well completion or abandonment. At a minimum, the form shall contain the following information:

(1) Name and address of facility/owner;

(2) Surveyed or global positioning system location, in latitude and longitude or Universal Transverse Mercator coordinates, of monitoring well(s) on a scaled map or plat;

(3) Driller and certification number;

(4) Date drilled;

(5) Driller's or Geologist's log;

(6) Total depth;

(7) Screened interval;

(8) Diameter and construction details;

(9) Depth to groundwater with date and time measured;

(10) Surveyed elevation of measuring point with respect to an established benchmark.

(11) Monitoring well approval number issued by the Department.

g. Monitoring wells constructed and reported to satisfy permitting or other regulatory requirements are not required to submit duplicate reports under this regulation.

h. Any well that acts as a source of contamination shall be repaired or permanently abandoned immediately after receipt of notice from the Department.

2. Additional Requirements for Permanent Conventionally Installed Monitoring Wells

a. Grouting.

(1) These monitoring wells shall be grouted from the top of the bentonite seal to the land surface.

(2) Grout is to be composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.

(a) Neat cement grout shall be composed of Class A, Type I Portland Cement mixed with not more than seven (7) gallons of clean water per bag (one cubic

foot or 94 pounds) of cement with a density of 15 to 16 pounds per gallon, or to manufacturer's specifications

(b) Bentonite-cement grout shall be composed of powdered bentonite (less than 5% by weight) mixed at not more than 8 gallons of water to the bag, with a density of 14 to 15 pounds per gallon, or to manufacturer's specifications.

(c) High solids sodium bentonite grout shall have a minimum of 20% solids and be mixed per manufacturer's specifications with water and/or other required additives.

(3) The diameter of the drilled hole shall be large enough to allow for a minimum of 1.5 inches of annular space on all sides of the casing for forced injection of grout through a tremie pipe.

(4) All grouting shall be accomplished using forced injection to emplace the grout. When emplacing the grouting material, the tremie pipe shall be lowered to the bottom of the zone to be grouted. The tremie pipe shall be kept full continuously from start to finish of the grouting procedure, with the discharge end of the tremie pipe being continuously submerged in the grout until the zone to be grouted is completely filled.

(5) A cement or aggregate reinforced concrete pad at the ground surface of appropriate durability and strength, considering the setting and location of each well, that extends six inches beyond the borehole diameter and six inches below ground surface is required. The pad shall be capable of preventing infiltration between the surface casing and the borehole to the subsurface.

b. Construction and Materials

(1) Casing shall be of sufficient strength to withstand normal forces encountered during and after well installation and be composed of material so as to minimally affect water quality analyses.

(2) Casing shall have a sufficient diameter to provide access for sampling equipment.

(3) A properly hydrated bentonite seal with a minimum thickness of twelve inches directly above the filter pack shall be used, if the well has a filter pack.

(4) The monitoring well intake or screen design shall minimize formational materials from entering the well. The filter pack

shall be utilized opposite the well screen as appropriate so that parameter analyses will be minimally affected.

(5) A locking cap or other security devices to prevent damage and/or vandalism shall be used.

(6) Monitoring wells completed below grade shall be in a watertight vault with a well cap to prevent infiltration of surface water into the well.

c. Well Identification.

All monitoring wells shall be properly labeled with an identification plate immediately upon well completion. The identification plate shall be constructed of a durable, weatherproof, rustproof, material. The identification plate shall be permanently secured to the well casing or enclosure floor around the casing where it is readily visible. The identification plate shall be permanently marked to show:

(1) Company name and certification number of the driller who installed the well;

(2) Date well was completed;

(3) Total depth (feet);

(4) Casing depth (feet);

(5) Screened interval;

(6) Designator and/or identification number.

d. Development.

Development shall be complete when the well produces water typical of the aquifer being utilized.

e. Abandonment

Abandonment shall be by forced injection of grout or pouring through a tremie pipe starting at the bottom of the well and proceeding to the surface in one continuous operation. The well shall be filled with either with neat cement, bentonite-cement, or 20% high solids sodium bentonite grout, from the bottom of the well to the land surface.

3. Additional Requirements for Permanent Direct Push Monitoring Wells

a. Direct Push Wells cannot be installed below a confining layer unless it can be demonstrated to the satisfaction of the Department that cross-contamination of the aquifer systems can be prevented.

b. Grouting.

(1) These monitoring wells shall be grouted from the top of the bentonite seal to the land surface.

(2) Grout is to be composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.

(a) Neat cement grout shall be composed of Class A, Type I Portland Cement mixed with not more than seven (7) gallons of clean water per bag (one cubic foot or 94 pounds) of cement with a density of 15 to 16 pounds per gallon, or to manufacturer's specifications

(b) Bentonite-cement grout shall be composed of powdered bentonite (less than 5% by weight) mixed at not more than 8 gallons of water to the bag, with a density of 14 to 15 pounds per gallon, or to manufacturer's specifications.

(c) High solids sodium bentonite grout shall have a minimum of 20% solids and be mixed per manufacturer's specifications with water and/or other required additives.

(3) The diameter of the annular space shall be large enough to allow for forced injection of grout through a tremie pipe.

(4) All grouting shall be accomplished using forced injection to emplace the grout. When emplacing the grouting material, the tremie pipe shall be lowered to the bottom of the zone to be grouted. The tremie pipe shall be kept full continuously from start to finish of the grouting procedure, with the discharge end of the tremie pipe being continuously submerged in the grout until the zone to be grouted is completely filled.

(5) A cement or aggregate reinforced concrete pad at the ground surface of appropriate durability and strength, considering the setting and location of each well, that extends six inches beyond the borehole diameter and six inches below ground surface is required. The pad shall be capable of preventing infiltration between the surface casing and the borehole to the subsurface.

c. Construction and Materials

(1) Casing shall be of sufficient strength to withstand normal forces encountered during and after well installation and be composed of material so as to minimally affect water quality analyses.

(2) Casing shall have a sufficient diameter to provide access for sampling equipment.

(3) The monitoring well intake or screen design shall minimize formational materials from entering the well. The well screen or intake shall be designed so that parameter analyses will be minimally affected.

(4) A locking cap or other security devices to prevent damage and/or vandalism shall be used.

(5) Monitoring wells completed below grade shall be in a watertight vault with a well cap to prevent infiltration of surface water into the well.

d. Well Identification.

All monitoring wells shall be properly labeled with an identification plate immediately upon well completion. The identification plate shall be constructed of a durable, weatherproof, rustproof, material. The identification plate shall be permanently secured to the well casing or enclosure floor around the casing where it is readily visible. The identification plate shall be permanently marked to show:

(1) Company name and certification number of the driller who installed the well;

(2) Date well was completed;

(3) Total depth (feet);

(4) Casing depth (feet);

(5) Screened Interval;

(6) Designator and/or identification number.

e. Development.

Development shall be complete when the well produces water typical of the aquifer being utilized.

f. Abandonment

(1) Permanent Direct Push Wells that do not penetrate a confining layer shall be abandoned by removing all casing from the subsurface and be grouted by forced injection through a tremie pipe from the total depth to the land surface, or by forced injection or pouring of neat cement, bentonite-cement, or 20% high solids sodium bentonite grout through a tremie pipe starting at the bottom of the well and proceeding to the surface in one continuous operation.

(2) Direct Push Wells that penetrate a confining layer shall be abandoned by forced injection or pouring of neat cement, bentonite-cement, or 20% high solids sodium bentonite grout through a tremie pipe starting at the bottom of the well and proceeding to the surface in one continuous operation.

4. Additional Requirements For Temporary Monitoring Wells

a. Construction and Materials

(1) Casing shall be of sufficient strength to withstand normal forces encountered during and after well installation and be

composed of material so as to minimally affect water quality analyses.

(2) Casing shall have a sufficient diameter to provide access for sampling equipment.

(3) The monitoring well intake or screen design shall minimize formational materials from entering the well. The filter pack or intake shall be utilized opposite the well screen as appropriate so that parameter analyses will be minimally affected.

b. Operation and Maintenance

All temporary monitoring wells shall be sealed with a watertight cap or seal until abandoned. Temporary monitoring wells shall be maintained such that they are not a source or channel of contamination before they are abandoned.

c. Abandonment.

(1) All temporary monitoring wells shall be abandoned within 5 days of borehole completion.

(2) A conventionally drilled temporary well shall be abandoned by forced injection of neat cement, bentonite-cement, or 20% high solids sodium bentonite grout through a tremie pipe starting at the bottom of the well and proceeding to the surface in one continuous operation.

(3) A Temporary Direct Push Well that does not penetrate a confining layer shall be abandoned by forced injection of neat cement, bentonite-cement, or 20% high solids sodium bentonite grout through a tremie pipe after the sampling device has been removed.

(4) A Temporary Direct Push Well that penetrates a confining layer shall be abandoned by forced injection of neat cement, bentonite-cement, or 20% high solids sodium bentonite grout through the sampling device as the sampling device is removed from the sub-surface. Abandonment shall occur during the initial withdrawal from the original push borehole and not by a separate tremie tool after the sampling device has been removed to ensure the breech in the confining layer is permanently sealed.

5. Use of Water Wells as Monitoring Wells.

Due to the variability involved, the use of a potable water well as a monitoring well shall be approved by the Department on a case-by-case basis.

6. Non-Standard Monitoring Wells

Due to the variability involved, the use of construction methods, techniques, or monitoring well designs not covered in this regulation shall be approved by the Department on a case-by-case basis.

I. BORINGS.

1. Geotechnical Borings.

    a. Geotechnical borings shall be abandoned within five days of borehole completion.

    b. Geotechnical borings shall be backfilled with a suitable material, such as the native material removed from the boring, to eliminate safety hazards and infiltration of runoff into the boring.

2. Exploration Borings

    a. Exploration borings shall be abandoned or cased to observe groundwater levels within five days of borehole completion or completion of geophysical logging. When cased, the casing shall be grouted per the requirements of R.61-71.F.2. and shall be capped for safety when not in use.

    b. Exploration borings in near surface unconsolidated formations or saprolite shall be backfilled with a suitable material, such as the native material removed from the boring, to eliminate safety hazards and infiltration of runoff into the boring.

    c. Exploration borings in indurated or consolidated formations shall be abandoned by filling with either bentonite-cement, neat cement, 20% high solids sodium bentonite grout, sand, or gravel to within no less than twenty feet of the surface and the remainder shall be filled with neat cement, bentonite-cement, or 20% high solids sodium bentonite grout.

    d. Exploration borings in indurated or consolidated formations shall be abandoned by forced injection of grout or pouring through a tremie pipe starting at the bottom of the borehole and proceeding to the surface in one continuous operation.

    e. Special precautions shall be used to abandon exploration borings if natural voids, such as solution cavities in limestone, significant fractures, and shear zones, are encountered. Suitable grout and grouting methods shall be used when such conditions exist to ensure the long-term integrity of the grout in the abandoned borehole.

    f. Exploration borings constructed on property that is included in an active mine permit shall be exempt from this Section of this regulation.

    g. A form provided and/or approved by the Department shall be completed and submitted to the Department within 30 days after completion of the exploration project, or phase in exploration drilling, whichever occurs first. The Department shall treat the approved form

completed and submitted for exploration boring abandonment as confidential trade secrets and proprietary business information and not subject to public disclosure under the Freedom of Information Act.

3. Environmental Soil Sampling Borings

   a. All analytical data obtained from each environmental soil sampling boring shall be submitted to the Department within 30 days of receipt of laboratory results unless another schedule has been approved by the Department.

   b. The boring shall be abandoned within five days of borehole completion.

   c. Borings five feet in depth or shallower may be abandoned by backfilling with native fill material.

   d. Borings greater than five feet in depth shall be completely filled from the bottom of the borehole to the land surface with bentonite-cement, neat cement, or 20% high solids sodium bentonite grout.

   e. The boring shall be abandoned by forced injection of grout or pouring through a tremie pipe starting at the bottom of the borehole and proceeding to the surface in one continuous operation.

   f. A Water Well Record Form 1903 or other form provided and/or approved by the Department shall be completed and submitted to the Department within 30 days after abandonment.

4. Horizontal Borings.

   a. Horizontal borings shall be backfilled with a suitable material, such as the native material removed from the boring, to eliminate safety hazards and infiltration of runoff into the boring.

J. GEOTHERMAL SYSTEM WELLS.

1. Drilling.

   a. The drilling process or use of drilling fluid additives shall not contaminate any aquifer.

   b. Water used in the drilling process shall be obtained from a source that will not result in chemical or biological contamination of any aquifer. Water taken directly from ponds, lakes, streams or other surface water sources shall not be used.

   c. Geothermal system wells shall not connect aquifers or zones that have documented differences:

      (1) In water quality that would result in contamination of any aquifer or zone such that any State Primary Drinking Water standard is exceeded;

(2) In static water levels that would result in depletion of water from any aquifer or zone, or significant loss of head in any aquifer or zone.

2. Grouting.

a. If the geothermal system well is to be cased, the diameter of the drilled hole shall be large enough to allow for a minimum of 1.5 inches of annular space on all sides of the casing for forced injection of grout through a tremie pipe. Grout shall fill the entire annular space from a minimum depth of twenty feet from the land surface at the time of well completion, unless otherwise approved by the Department.

b. If the geothermal system well will not be cased, the borehole may be filled with either bentonite-cement, neat cement, 20% high solids sodium bentonite grout, sand, or gravel to no closer than twenty feet below the ground surface. The remaining twenty feet to the ground surface shall be filled with neat cement, bentonite-cement, or 20% high solids sodium bentonite grout.

c. Grout is to be composed of neat cement, a bentonite cement mixture, or high solids sodium bentonite grout.

(1) Neat cement grout shall be composed of Class A, Type I Portland Cement mixed with not more than seven (7) gallons of clean water per bag (one cubic foot or 94 pounds) of cement with a density of 15 to 16 pounds per gallon, or to manufacturer's specifications.

(2) Bentonite-cement grout shall be composed of powdered bentonite (less than 5% by weight) mixed at not more than 8 gallons of water to the bag, with a density of 14 to 15 pounds per gallon, or to manufacturer's specifications.

(3) High solids sodium bentonite grout shall have a minimum of 20% solids and be mixed per manufacturer's specifications with water and/or other required additives.

d. All grouting shall be accomplished using forced injection to emplace the grout. When emplacing the grouting material, the tremie pipe shall be lowered to the bottom of the zone to be grouted. The tremie pipe shall be kept full continuously from start to finish of the grouting procedure, with the discharge end of the tremie pipe being continuously submerged in the grout until the zone to be grouted is completely filled.

e. Geothermal system wells shall be grouted in-place within five (5) days after borehole completion.

f. When high solids sodium bentonite grouts are used, a vapor barrier at the land surface at least the width of the annular space made of suitable materials, as approved by the Department, such as native soils, gravel,

sand, or thermoplastic material, is required for public safety and structural stability of the well.

3. Well Identification.

These wells shall be properly labeled with an identification plate immediately upon well completion. The identification plate shall be constructed of a durable, weatherproof, rustproof, material. The identification plate shall be permanently secured to the well casing or enclosure floor around the casing where it is readily visible. The identification plate shall be permanently marked to show:

a. Company name and certification number of the driller who installed the well;

b. Date well was completed;

c. Total depth (feet); and

d. Casing depth (feet).

4. Reporting.

The Water Well Record Form 1903, or other approved form, shall be completed and submitted to the Department by the contractor within thirty days after well completion.

K. VIOLATIONS AND PENALTIES.

1. Violations of this regulation shall be subject to penalties as provided in Sections 48-1-320, 44-55-90 and 48-1-330 of the 1976 S.C. Code of Laws.

2. Whenever the Department finds that a well driller is in violation of a requirement under this regulation, the Department will issue a written notice of violation with a requirement to correct all violations within a specified time period. Whether the noted violation(s) is properly corrected within the specified time period, the extent of deviation from the regulation, the potential for harm, and the historical record of violations by that well driller, shall be considered by the Department in determining additional enforcement actions, if any, and any associated penalties.

3. Should a violation be determined by the Department to have occurred despite the well driller following all applicable regulations and manufacturer's specifications, the Department shall direct the well driller to correct the noted violation within a specified time period. If this violation is corrected within the specified time period, no monetary penalty will be assessed for that violation.

4. After written notice of violation, the Department may issue an order or commence an action in court requiring the well driller to comply with the permit, regulation, standard, or requirement, or may request the Attorney General to commence an action under this subsection in the appropriate court. The Department may also assess civil penalties as provided in this section for violations of the provisions of this regulation or for violating any order, permit, regulation, or standard.

5. A well driller who fails to take appropriate corrective action, after receiving written notice of violation of a provision of this regulation, is liable for civil penalties or criminal prosecution.
6. The Department shall have full access to a well driller's bond as required by the Department of Labor, Licensing, and Regulation to correct a violation of this regulation where, as part of a Department enforcement action, a well driller is unwilling or unable to take required corrective actions.
7. If the Department finds a person not certified by the Department of Labor, Licensing, and Regulation in the act of drilling a well, the Department may issue an order or field citation requiring the person to immediately cease and desist operations, or seek a court order enjoining further drilling.

L. SEVERABILITY.

Should any section, paragraph, sentence, clause, phrase, or other part of this regulation be declared invalid for any reason, the remainder shall not be affected thereby.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Packing, Marking, Labeling and Shipping of Environmental and Waste Samples**

Effective Date: April 20, 2011	**Number:** SESDPROC-209-R2

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-209-R2, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R1. **Title Page:** Changed EIB Branch Chief from Antonio Quinones to Archie Lee. Changed Field Quality Manager from Laura Ackerman to Liza Montalvo. **Revision History:** Previous versions of the document will be maintained by the Document Control Coordinator (DCC). Changed from Field Quality Manager to DCC. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance. Deleted reference to the H: drive. **Section 3, fifth paragraph**: On item #2 on the list, replaced "be sure" with "ensure that." On item #3, deleted the last sentence that stated that only "Up to three VOA vials may be packed in one Whirl-Pak container or bag." Deleted outdated item #4 referencing shipment of VOA vials in metal cans. On items #5 and #6, removed references to vermiculite in favor of generic "absorbent material."	April 20, 2011
SESDPROC-209-R1, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R0. **General** Corrected any typographical, grammatical and/or editorial errors. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief.	November 1, 2007

Section 1.3 Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.4** Alphabetized and revised the referencing style for consistency. Added reference to the SHEMP Procedures and Policy Manual. **Section 1.5.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.	
SESDPROC-209-R0, Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by Department of Transportation under 49 CFR, Subchapter C, Hazardous Materials Regulations, and the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000. This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when packing, marking, labeling and shipping environmental and waste samples to ensure that all shipments are in compliance with the above regulations and guidance.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when packing, marking, labeling, and shipping environmental samples and dangerous goods by air transport. Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials (dangerous goods) samples.

In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials. Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods.

Government employees transporting samples or hazardous materials (i.e., preservatives or waste samples) in government vehicles are not subject to the requirements of this section in accordance with 49 CFR 171.1(d)(5). EPA contractors, however, are not covered by this exemption and may not transport these materials without full compliance with 49 CFR.

Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

Title 40 Code of Federal Regulations (CFR), Pt. 136.3, Identification of Test Procedures, July 1, 2001. See Table II, Footnote 3.

Title 49 CFR, Pt. 171.1(d)(5), Applicability of Hazardous Materials Regulations (HMR) to Persons and Functions.

United States Department of Transportation (US DOT). 2003. Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No. 02-0093, February 13, 2003.

US Environmental Protection Agency (US EPA) Order 1000.18, February 16, 1979.

US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when packing, marking, labeling, and shipping environmental or waste samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional.

2 Shipment of Dangerous Goods

The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. ***Dangerous goods must not be offered for air transport by any personnel except SESD's dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment.***

3 Shipment of Environmental Samples

Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples". By this memorandum, the shipment of the following unpreserved samples is not regulated:

- Drinking water
- Treated effluent
- Biological specimens
- Sediment
- Water treatment plant sludge
- POTW sludge

In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 or the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. This provision is also discussed in correspondence between DOT and EPA (Department of Transportation, Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No.: 02-0093, February 13, 2003). It is the shippers' (individual signing the air waybill) responsibility to ensure that proper amounts of preservative are used:

- Drinking water
- Ambient water
- Treated effluent
- Biological specimens
- Sediment
- Wastewater treatment plant sludge
- Water treatment plant sludge

Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between USEPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

Untreated wastewater and sludge from Publicly Owned Treatment Works (POTWs) are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

Environmental samples should be packed prior to shipment by air using the following procedures:

1. Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).

2. Ensure that the lids on all bottles are tight (will not leak).

3. Place bottles in separate and appropriately sized polyethylene bags and seal the bags. If available, the use of Whirl-Pak bags is preferable, if unavailable seal regular bags with tape (plastic electrical tape).

4. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy duty plastic bag.

5. Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.

6. Put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with absorbent material.

7. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

8. Place the Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) into a plastic bag, and tape the bag to the inner side of the cooler lid.

9. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Soil Sampling**

Effective Date: December 20, 2011	**Number:** SESDPROC-300-R2

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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-300-R2, *Soil Sampling*, replaces SESDPROC-300-R1. **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Updated the Enforcement and Investigations Branch Chief to Archie Lee, and the Field Quality Manager to Bobby Lewis. **Revision History:** On the third sentence, replaced Field Quality Manager with Document Control Coordinator. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Revised the last sentence to reflect that the official copy of this procedure resides on the SESD local area network (LAN), and that the Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN. **Section 1.4:** Unused references removed. Reference to Method 5035 added. Alphabetized. **Section 1.5.2:** Added bullets #8 and #9. (Bullet #8 – Discussion of sampling in landscaped areas. Bullet #9 – Discussion of sampling in non-landscaped areas.) **Section 2:** The discussion of Method 5035 has been moved to its own Section (Section 3) for clarity. Subsequent Sections were re-numbered. **Section 4.1:** Added paragraphs #2 and #3. (The information on these paragraphs originally appeared as bullets #2 and #3 in Section 4.2.1.) In addition, determining the initial sampling depth is now defined in paragraph #2. **Section 4.2.1:** As mentioned above, deleted bullets #2 and #3 from this Section and moved this information to paragraphs #2 and #3 in Section 4.1. **Section 5.1:** On paragraph #1, rewrote sentences #2 and #3 for clarity. Added the last sentence. Added paragraph #2.	December 20, 2011

Section 5.3: Rewrote the last sentence in the paragraph to clarify that, "although the Macro-Core® sampler can be used as an open-barrel sampler, in SESD usage, the piston point is always used to prevent the collection of slough from the borehole sides." **Section 5.5:** Rewrote bullet #1 to clarify acceptable materials for liners. Rewrote bullet #3 (Core Catchers) to clarify that, given the limited sample contact that core-catchers have with the sample material, most standard core-catchers available for a tool system will be acceptable. Added bullets #4 (Decontamination) and #5 (Decommissioning). **Section 6.1:** Added paragraph #2 to this section. (Determining the initial sampling depth is now defined.) **Section 7.1:** Added paragraph #2 to this section. (Determining the initial sampling depth is now defined.) **Section 8.1:** Added paragraph #2 to this section. (Determining the initial sampling depth is now defined.)	
SESDPROC-300-R1, *Soil Sampling*, replaces SESDPROC-300-R0. **General** Corrected any typographical, grammatical and/or editorial errors. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. **Section 1.3** Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.4** Updated referenced operating procedures due to changes in title names. Alphabetized and revised the referencing style for consistency. **Section 1.5.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual. **Section 1.5.2, 4th bullet** Added references to the CFR and IATA's Dangerous Goods Regulations. **Section 2.7** Updated referenced operating procedures due to changes in title names.	November 1, 2007
SESDPROC-300-R0, Soil Sampling, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting soil samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling soil samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a soil sample, the variant procedure will be documented in the field logbook and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN, and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field X-Ray Fluorescence (XRF) Measurement, SESDPROC-107, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

US EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Most Recent Version (Method 5035)

US EPA. Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting soil samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting soil samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.

- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader in the project files.
- Sampling in landscaped areas: When sampling in landscaped areas, cuttings should be placed on plastic sheeting and returned to the borehole upon completion of the sample collection. Any 'turf plug' generated during the sampling process should be returned to the borehole.
- Sampling in non-landscaped areas: Return any unused sample material back to the auger, drill or push hole from which the sample was collected.

2 Special Sampling Considerations

2.1 Special Precautions for Trace Contaminant Soil Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different sample is collected and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be collected, handled and stored separately.
- All background samples shall be segregated from obvious high-concentration or waste samples. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly-contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other member(s) collect the samples.
- Samplers must use new, verified/certified-clean disposable or non-disposable equipment cleaned according to procedures contained in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205), for collection of samples for trace metals or organic compound analyses.

2.2 Sample Homogenization

1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 4.

2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same approximate volume.

3. All soil samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. ***Samples for VOC analysis are not homogenized.*** 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 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. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4. Place the sample into an appropriate, labeled container(s) by using the alternate shoveling method and secure the cap(s) tightly. The alternate shoveling method involves placing a spoonful of soil in each container in sequence and repeating until the containers are full or the sample volume has been exhausted. Threads on the container and lid should be cleaned to ensure a tight seal when closed.

2.3 Dressing Soil Surfaces

Any time a vertical or near vertical surface is sampled, such as achieved when shovels or similar devices are used for subsurface sampling, the surface should be dressed (scraped) to remove smeared soil. This is necessary to minimize the effects of contaminant migration interferences due to smearing of material from other levels.

2.4 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same soil type. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools. SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) contains other procedures that may be applicable to soil sampling investigations.

2.5 Records

Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation, as described in the SESD Operating Procedure for Logbooks (SESDPROC-010) and the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

3 Method 5035

The procedures outlined here are summarized from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Method 5035.*

3.1 Soil Samples for Volatile Organic Compounds (VOC) Analysis

If samples are to be analyzed for volatile organic compounds, they should be collected in a manner that minimizes disturbance of the sample. For example, when sampling with an auger bucket, the sample for VOC analysis should be collected directly from the auger bucket (preferred) or from minimally disturbed material immediately after an auger bucket is emptied into the pan. The sample shall be containerized by filling an En Core® Sampler or other Method 5035 compatible container. ***Samples for VOC analysis are not homogenized.*** Preservatives may be required for some samples with certain variations of Method 5035. Consult the method or the principal analytical chemist to determine if preservatives are necessary.

3.2 Soil Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of volatile organic compounds (VOCs) in soils at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

3.2.1 Equipment

Soil for VOC analyses may be retrieved using any of the SESD soil sampling methods described in Sections 4 through 8 of this procedure. Once the soil has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2-oz. soil VOC container, or pre-prepared 40 mL vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various sub-sampling methods are described below.

3.2.2 Sampling Methodology - Low Concentrations (<200 µg/kg)

When the total VOC concentration in the soil is expected to be less than 200 µg/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 mL pre-prepared vial) immediately to reduce volatilization losses. The 40 mL

vials should contain 10 mL of organic-free water for an un-preserved sample or approximately 10 mL of organic-free water and a preservative. It is recommended that the 40 mL vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked.

A soil sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 mL pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. soil jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 mL pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE: After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Soil samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in the original foil bag. All foil bags containing En Core® samplers are then placed in a plastic bag and sealed with custody tape, if required.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-mL pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 mL containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

3.2.3 Sampling Methodology - High Concentrations (>200 µg/kg)

Based upon the data quality objectives and the detection level requirements, this high level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Soils\sediments containing high total VOC concentrations may also be collected as described in Section 3.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 mL methanol.

3.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved, as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size and the lack of sample mixing, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the Analytical Support Branch *Laboratory Operations and Quality Assurance Manual* (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC soil/sediment sample on ice is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Moisture and Preservative Compatibility (MOICA)

Samplers must ensure that the laboratory has sufficient material to determine percent moisture in the VOC soil/sediment sample to correct the analytical results to dry weight. If other analyses requiring percent moisture determination are

being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent moisture determination will be required. The sample collected for percent moisture may also be used by the laboratory to check for preservative compatibility.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities.

The summary table on the following page lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. SESD's goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect two 40 mL vials with ≈ 5 grams of sample, and one 2 oz. glass jar w/septum lid for screening, % moisture and preservative compatibility.	Screening conducted by lab.	Presently a 48-hour holding time for unpreserved samples. Sample containers must be tared.
2	Collect three En Core® samplers, and one 2 oz. glass jar w/septum lid for screening, % moisture and preservative compatibility.	Lab conducts all preservation/preparation procedures.	Presently a 48- hour holding time for preparation of samples.
3	Collect two 40 mL vials with 5 grams of sample and preserve w/methanol or sodium bisulfate, and one 2-oz. glass jar w/septum lid for screening, % moisture and preservative compatibility.	High level VOC samples may be composited. Longer holding time.	Hazardous materials used in the field. Sample containers must be tared.
4	Collect one 2-oz. glass jar w/septum lid for analysis, % moisture and preservative compatibility (high level VOC only).	Lab conducts all preservation/preparation procedures.	May have significant VOC loss.

4 Manual Soil Sampling Methods

4.1 General

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches; however, the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical.

If a thick, matted root zone, gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials.

When compositing, make sure that each composite location (aliquot) consist of equal volumes, i.e., same number of equal spoonfuls.

4.2 Spoons

Stainless steel spoons may be used for surface soil sampling to depths of approximately 6 inches below ground surface where conditions are generally soft and non-indurated, and there is no problematic vegetative layer to penetrate.

4.2.1 Special Considerations When Using Spoons

- When using stainless steel spoons, consideration must be given to the procedure used to collect the volatile organic compound sample. If the soil being sampled is cohesive and holds its in situ texture in the spoon, the En Core® Sampler or syringe used to collect the sub-sample for Method 5035 should be plugged directly from the spoon. If, however, the soil is not cohesive and crumbles when removed from the ground surface for sampling, consideration should be given to plugging the sample for Method 5035 directly from the ground surface at a depth appropriate for the investigation Data Quality Objectives.

4.3 Hand Augers

Hand augers may be used to advance boreholes and collect soil samples in the surface and shallow subsurface intervals. Typically, 4-inch stainless steel auger buckets with cutting heads are used. The bucket is advanced by simultaneously pushing and turning using an attached handle with extensions (if needed).

4.3.1 Surface Soil Sampling

When conducting surface soil sampling with hand augers, the auger buckets may be used with a handle alone or with a handle and extensions. The bucket is advanced to the appropriate depth and the contents are transferred to the homogenization container for processing. Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

4.3.2 Subsurface Soil Sampling

Hand augers are the most common equipment used to collect shallow subsurface soil samples. Auger holes are advanced one bucket at a time until the sample depth is achieved. When the sample depth is reached, the bucket used to advance the hole is removed and a clean bucket is attached. The clean auger bucket is then placed in the hole and filled with soil to make up the sample and removed.

The practical depth of investigation using a hand auger depends upon the soil properties and depth of investigation. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow or collapse. Hand augers may also be of limited use in tight clays or cemented sands. In these soil types, the greater the depth attempted, the more difficult it is to recover a sample due to increased friction and torqueing of the hand auger extensions. At some point these problems become so severe that power equipment must be used.

4.3.3 Special Considerations for Soil Sampling with the Hand Auger

- Because of the tendency for the auger bucket to scrape material from the sides of the auger hole while being extracted, the top several inches of soil in the auger bucket should be discarded prior to placing the bucket contents in the homogenization container for processing.
- Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035. Collect the VOC sample directly from the auger bucket, if possible.
- Power augers, such as the Little Beaver® and drill rigs may be used to advance boreholes to depths for subsurface soil sampling with the hand auger. They may not be used for sample collection. When power augers are used to advance a borehole to depth for sampling, care must be taken that exhaust fumes, gasoline and/or oil do not contaminate the borehole or area in the immediate vicinity of sampling.
- When moving to a new sampling location, the entire hand auger assembly must be replaced with a properly decontaminated hand auger assembly.

5 Direct Push Soil Sampling Methods

5.1 General

These methods are used primarily to collect shallow and deep subsurface soil samples. Three samplers are available for use within the Division's direct push tooling inventory. All of the sampling tools involve the collection and retrieval of the soil sample within a thin-walled liner. The following sections describe each of the specific sampling methods that can be accomplished using direct push techniques, along with details specific to each method. While SESD currently uses the sample tooling described, tooling of similar design and materials is acceptable.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

5.2 Large Bore® Soil Sampler

The Large Bore® (LB) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of depth-discrete subsurface soil samples. The sample barrel is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The LB® sampler is capable of recovering a discrete sample core 22 inches x 1.0 inch (559 mm x 25 mm) contained inside a removable liner. The resultant sample volume is a maximum of 283 mL.

After the LB® sample barrel is equipped with the cutting shoe and liner, the piston-rod point assembly is inserted, along with the drive head and piston stop assembly. The assembled sampler is driven to the desired sampling depth, at which time the piston stop pin is removed, freeing the push point. The LB® sampler is then pushed into the soil a distance equal to the length of the LB® sample barrel. The probe rod string, with the LB® sampler attached, is then removed from the subsurface. After retrieval, the LB® sampler is then removed from the probe rod string. The drive head is then removed to allow removal of the liner and soil sample.

5.3 Macro-Core® Soil Sampler

The Macro-Core® (MC) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of either continuous or depth-discrete subsurface soil samples. Although other lengths are available, the standard MC® sampler has an assembled length of approximately 52 inches (1321 mm) with an outside diameter of 2.2 inches (56 mm). The MC® sampler is capable of recovering a discrete sample core 45 inches x 1.5 inches (1143 mm x 38 mm) contained inside a removable liner. The resultant sample volume is a maximum of 1300 mL. The MC® sampler may be used in either an open-tube or closed-point configuration. Although the

MC® sampler can be used as an open-barrel sampler, in SESD usage, the piston point is always used to prevent the collection of slough from the borehole sides.

5.4 Dual Tube Soil Sampling System

The Dual Tube 21 soil sampling system is a direct push system for collecting continuous core samples of unconsolidated materials from within a sealed outer casing of 2.125-inch (54 mm) OD probe rod. The samples are collected within a liner that is threaded onto the leading end of a string of 1.0-inch diameter probe rod. Collected samples have a volume of up to 800 mL in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core. Use of this method allows for collection of continuous core inside a cased hole, minimizing or preventing cross-contamination between different intervals during sample collection. The outer casing is advanced, one core length at a time, with only the inner probe rod and core being removed and replaced between samples. If the sampling zone of interest begins at some depth below ground surface, a solid drive tip must be used to drive the dual tube assembly and core to its initial sample depth.

5.5 Special Considerations When Using Direct Push Sampling Methods

- *Liner Use and Material Selection* – Direct Push Soil Samples are collected within a liner to facilitate removal of sample material from the sample barrel. The liners may only be available in a limited number of materials for a given sample tool, although overall, liners are available in brass, stainless steel, cellulose acetate butyrate (CAB), polyethylene terepthalate glycol (PETG), polyvinyl chloride (PVC) and Teflon®. For most SESD investigations, the standard polymer liner material for a sampling tool will be acceptable. When the study objectives require very low reporting levels or unusual contaminants of concern, the use of more inert liner materials such as Teflon® or stainless steel may be necessary.

- *Sample Orientation* – When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely the depth at which an aliquot was collected. Maintaining proper orientation is typically accomplished using vinyl end caps. Convention is to place red caps on the top of the liner and black caps on the bottom to maintain proper sample orientation. Orientation can also be indicated by marking on the exterior of the liner with a permanent marker.

- *Core Catchers* – Occasionally the material being sampled lacks cohesiveness and is subject to crumbling and falling out of the sample liner. In cases such as these, the use of core catchers on the leading end of the sampler may help retain the sample until it is retrieved to the surface. Core catchers may only be available in specific materials and should be evaluated for suitability.

However, given the limited sample contact that core-catchers have with the sample material, most standard core-catchers available for a tool system will be acceptable.

- *Decontamination* – The cutting shoe and piston rod point are to be decontaminated between each sample, using the procedures specified for the collection of trace organic and inorganic compounds found in Field Equipment and Decontamination – SESDPROC-205, most recent version. Within a borehole, the sample barrel, rods, and drive head may be subjected to an abbreviated cleaning to remove obvious and loose material, but must be cleaned between boreholes using the procedures specified for downhole drilling equipment in Field Equipment and Decontamination – SESDPROC-205, most recent version.

- *Decommissioning* – Boreholes must be decommissioned after the completion of sampling. Boreholes less than 10 feet depth that remain open and do not approach the water table may be decommissioned by pouring 30% solids bentonite grout from the surface, or pouring bentonite pellets from the surface, hydrating the pellets in lifts. Boreholes deeper than 10 feet, or any borehole that intercepts groundwater, must be decommissioned by pressure grouting with 30% solids bentonite grout, either through a re-entry tool string or through tremie pipe introduced to within several feet of the borehole bottom.

- *VOC Sample Collection* – Observe precautions for volatile organic compound sample collection found in Section 3 of this procedure.

6 Split Spoon/Drill Rig Methods

6.1 General

Split spoon sampling methods are used primarily to collect shallow and deep subsurface soil samples. All split spoon samplers, regardless of size, are basically split cylindrical barrels that are threaded on each end. The leading end is held together with a beveled threaded collar that functions as a cutting shoe. The other end is held together with a threaded collar that serves as the sub used to attach the spoon to the string of drill rod. Two basic methods are available for use, including the smaller diameter standard split spoon, driven with the drill rig safety hammer, and the larger diameter continuous split spoon, advanced inside and slightly ahead of the lead auger during hollow stem auger drilling. The following sections describe each of the specific sampling methods, along with details specific to each method.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

6.2 Standard Split Spoon

A drill rig is used to advance a borehole to the target depth. The drill string is then removed and a standard split spoon is attached to a string of drill rod. Split spoons used for soil sampling must be constructed of stainless steel and are typically 2.0-inches OD (1.5-inches ID) and 18-inches to 24-inches in length. Other diameters and lengths are common and may be used if constructed of the proper material. After the spoon is attached to the string of drill rod, it is lowered into the borehole. The safety hammer is then used to drive the split spoon into the soil at the bottom of the borehole. After the split spoon has been driven into the soil, filling the spoon, it is retrieved to the surface, where it is removed from the drill rod string and opened for sample acquisition.

6.3 Continuous Split Spoon

The continuous split spoon is a large diameter split spoon that is advanced into the soil column inside a hollow stem auger. Continuous split spoons are typically 3 to 5 inches in diameter and either 5 feet or 10 feet in length, although the 5-foot long samplers are most common. After the auger string has been advanced into the soil column a distance equal to the length of the sampler being used it is returned to the surface. The sampler is removed from inside the hollow stem auger and the threaded collars are removed. The split spoon is then opened for sampling.

6.4 Special Considerations When Using Split Spoon Sampling Methods

- Always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the borehole wall after removal of the drill string prior to and during inserting the split spoon.
- Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

7 Shelby Tube/Thin-Walled Sampling Methods

7.1 General

Shelby tubes, also referred to generically as thin-walled push tubes or Acker thin-walled samplers, are used to collect subsurface soil samples in cohesive soils and clays during drilling activities. In addition to samples for chemical analyses, Shelby tubes are also used to collect relatively undisturbed soil samples for geotechnical analyses, such as hydraulic conductivity and permeability, to support hydrogeologic characterizations at hazardous waste and other sites.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with this device.

7.2 Shelby Tube Sampling Method

A typical Shelby tube is 30 inches in length and has a 3.0-inch OD (2.875-inch ID) and may be constructed of steel, stainless steel, galvanized steel, or brass. They also typically are attached to push heads that are constructed with a ball-check to aid in holding the contained sample during retrieval. If used for collecting samples for chemical analyses, it must be constructed of stainless steel. If used for collecting samples for standard geotechnical parameters, any material is acceptable.

To collect a sample, the tube is attached to a string of drill rod and is lowered into the borehole, where the sampler is then pressed into the undisturbed material by hydraulic force. After retrieval to the surface, the tube containing the sample is then removed from the sampler head. If samples for chemical analyses are needed, the soil contained inside the tube is then removed for sample acquisition. If the sample is collected for geotechnical parameters, the tube is typically capped, maintaining the sample in its relatively undisturbed state, and shipped to the appropriate geotechnical laboratory.

7.3 Special Considerations When Using Split Spoon Sampling Methods

Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

8 Backhoe Sampling Method

8.1 General

Backhoes may be used in the collection of surface and shallow subsurface soil samples. The trenches created by excavation with a backhoe offer the capability of collecting samples from very specific intervals and allow visual correlation with vertically and horizontally adjacent material. If possible, the sample should be collected without entering the trench. Samples may be obtained from the trench wall or they may be obtained directly from the bucket at the surface. The following sections describe various techniques for safely collecting representative soil samples with the aid of a backhoe.

The depth measurement for the sample begins at the top of the soil horizon.

8.2 Scoop-and-Bracket Method

If a sample interval is targeted from the surface, it can be sampled using a stainless steel scoop and bracket. First a scoop and bracket are affixed to a length of conduit and is lowered into the backhoe pit. The first step is to take the scoop and scrape away the soil comprising the surface of the excavated wall. This material likely represents soil that has been smeared by the backhoe bucket from adjacent material. After the smeared material has been scraped off, the original stainless steel scoop is removed and a clean stainless steel scoop is placed on the bracket. The clean scoop can then be used to remove sufficient volume of soil from the excavation wall to make up the required sample volume.

8.3 Direct-from-Bucket Method

It is also possible to collect soil samples directly from the backhoe bucket at the surface. Some precision with respect to actual depth or location may be lost with this method but if the soil to be sampled is uniquely distinguishable from the adjacent or nearby soils, it may be possible to characterize the material as to location and depth. In order to ensure representativeness, it is also advisable to dress the surface to be sampled by scraping off any smeared material that may cross-contaminate the sample.

8.4 Special Considerations When Sampling with a Backhoe

- Do not physically enter backhoe excavations to collect a sample. Use either procedure 8.2, Scoop-and-Bracket Method, or procedure 8.3, Direct-from-Bucket Method to obtain soil for sampling.
- Smearing is an important issue when sampling with a backhoe. Measures must be taken, such as dressing the surfaces to be sampled (see Section 2.3), to mitigate problems with smearing.

- Paint, grease and rust must be removed and the bucket decontaminated prior to sample collection.
- Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

Appendix B
Laboratory Supporting Information

STANDARD OPERATING PROCEDURE FOR

ULTRASONIC EXTRACTION FOR BASE/NEUTRAL AND ACID COMPOUNDS (BNA)

SW-846 3550C

Approved by: _____ Date: 4/11/14
Thomas Steiner, Extractions Supervisor

Approved by: _____ Date: 4/11/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 4/11/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This procedure is designed for the preparation of solid samples for the analysis of extractable base, neutral, and acid organic compounds.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Soil

3.0 SUMMARY OF TEST METHOD

3.1 A measured volume of sample, approximately 30 g, or a reduced volume based on sample matrix, is serially extracted with 1:1 methylene chloride/acetone using ultrasonic extraction.

3.2 The extract is dried, concentrated, and bottled for analysis of BNAs by GC/MS.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware and other sample extraction apparatus may yield interferences to sample analysis. All these must be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

6.4 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Organochlorine pesticides, phthalate esters and phenols may react under basic conditions. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Glass beakers – 250 mL

8.2 Kuderna-Danish (K-D) apparatus

- 8.2.1 Receiver, 10 mL graduated
- 8.2.2 Evaporation Flask, 500 mL - attached to the receiver with a blue clamp
- 8.2.3 3-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.3 2-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.4 Hot plate

8.5 Spatula, stainless steel or wooden stirrer.

8.6 1 mL pipette (verified daily before use) or 1 mL gas tight syringe

8.7 Funnels, glass or Teflon

8.8 Filter Paper – Whatman 41 18.5 cm or equivalent.

8.9 Boiling chips, Teflon or equivalent

8.10 Sonicator- Fisher Scientific Ultrasonic Dismembrator equipped with ¾ inch disruptor horn.

8.11 Pasteur pipets

8.12 Water bath – Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control (± 5°C). The bath must be used in a hood.

8.13 Analytical balance – capable of weighing to 0.1g and calibrated daily when in use

8.14 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps

8.15 Volumetric Flasks – 1.0 mL, 5.0 mL, 10.0 mL

8.16 Nitrogen Blow-Down Unit

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor.
Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized water

9.1.2 Sodium sulfate (ACS) – granular, anhydrous, heated to approximately 400 °C for a minimum of 4 hours in a shallow tray, cooled and stored in a glass containers with Teflon lined caps. Record in the Sodium Sulfate Logbook and assign a lot number. Label all glass containers of sodium sulfate with the batch number, the date baked, expiration date and the initials of the preparer.

9.1.3 Methylene chloride

9.1.4 Acetone

9.1.5 1:1 Methylene chloride/Acetone – purchased premixed.

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Surrogate Stock Standards (commercially prepared) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.1.1 Base/Neutral Surrogate Mix (5000 µg/mL)

9.2.1.2 Acid Surrogate Mix (10,000 µg/mL)

9.2.2 Surrogate Working Solution Mix (50/100 µg/mL) – add 10.0 mL of Base/Neutral Surrogate Mix solution and 10.0 mL of Acid Surrogate Mix solution to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months

from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.3 625 Stock Spiking Solution (commercially prepared, multi-analyte mix, 100 µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.4 625 Working Spiking Solution (50 µg/mL) – add 25 mL of 625 stock standard to 25 mL of acetone. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.5 Base Neutral Stock Spiking Solution (commercially prepared, multi-analyte mix, 5000 µg/mL) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.6 Acid Stock Spiking Solution (commercially prepared, multi-analyte mix, 7500 µg/mL) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.7 8270 Working Spiking Solution (100 µg/mL) – add 4.0 mL of Base/Neutral spiking solution and 2.68 mL of Acid spiking solution to a 200 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.8 APP9 Stock Spiking Solution (commercially custom prepared, multi-analyte mix, 400µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.9 APP9 Working Spiking Solution (100 µg/mL) – add 5.0 mL of APP9 stock spiking solution to a 20 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.10 Commercially prepared standards at the desired concentration or diluted with the appropriate solvent may have to be purchased based on the project or client specified analyte list. These standards will expire according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem. Store standards according to manufacturer's instructions.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 14 days from collection to extraction and 40 days from extraction to analysis

12.0 QUALITY CONTROL

12.1 One method blank, LCS, LCSD, MS, and MSD shall be prepared per batch, a maximum of twenty samples.

12.2 If insufficient sample is available for a MS and/or MSD then document this on the Extraction Preparation Form.

12.3 QC samples are prepared in the exact manner of the samples using the Procedure in Section 14.0.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified each day of use. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook associated with each pipette.

14.0 PROCEDURE

14.1 Rinse all 250 mL beakers with methylene chloride three times. Dispose of the rinsate in the appropriate solvent waste container.

14.2 Rinse a K-D apparatus three times with methylene chloride. Dispose of the rinsate in the appropriate solvent waste container.

14.3 Label each beaker and K-D apparatus with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.

14.4 With each extraction batch, a method blank, LCS, LCSD and MS/MSD are included. If insufficient sample is available for a MS and/or MSD, document on the Extraction Preparation Form.

14.5 Weigh a 30 g aliquot of sodium sulfate into a methylene chloride rinsed beaker labeled Blank; repeat for the LCS and LCSD. Record the weight on the Extraction Preparation Form.

14.6 Homogenize sample by pouring the contents of the sample container onto a piece of butcher paper and chop and mix with a spatula. Remove any material such as sticks, leaves, or rocks. Weigh 30 ± 0.5 g of homogenized sample to the nearest 0.1 g into the beaker. Record weight on the Extraction Preparation Form.

14.7 Add approximately 30 g of sodium sulfate to each sample and mix well. The sample should have a free flowing sandy texture at this point. If not, add Sodium Sulfate until the sample is free flowing.

14.8 Add 1.0 mL of the surrogate standard spiking solution to each beaker. Spike the LCS/LCSD and the MS/MSD beakers with 1.0 mL of the required 8270 or 625 spiking solution. If Appendix IX compounds are required, add 1.0 mL of the APP9 spiking solution. Note: if multiple spiking solutions are required, the LCS/LCSD and MS/MSD are prepared separately with the associated standards. Mix well. All extraction prep batches require a spike witness that is a peer technician, analyst or someone from supervisory or management level. Only employees that have received spike witness training can act as a spike witness. In the event that an employee is working outside of normal business hours and no qualified spike witness is available, then it is solely the individual technician's responsibility to verify all appropriate spikes and surrogates have been added. In this event, no spike witness will be indicated on the Extraction Preparation Form.

14.9 Immediately add approximately 100 mL of 1:1 methylene chloride/acetone. The solvent level should be about 2 inches above the sample in the beaker.

14.10 The sonicator is tested daily prior to use by pressing the test button on the front panel. If the sonicator is operating correctly the instrument returns to ready mode and is ready for use. If an error message is given, notify supervisor and do not sonicate samples using this sonicator.

14.11 Sonicate each sample and QC samples for 3 minutes by placing the probe of the sonicator about ½ inch below the surface of the solvent but above the sediment.

14.12 Decant and filter extracts through a funnel lined with folded filter paper into a labeled K-D apparatus.

14.13 Repeat the extraction two more times with two additional 100 mL portions of 1:1 methylene chloride/acetone. Combine the solvent from each extraction into the labeled K-D.

14.14 On the final sonication transfer the entire contents of the beaker into the funnel and rinse with methylene chloride.

14.15 Add one or two Teflon boiling chips to the labeled K-D apparatus. Rinse the 3 ball Snyder column with methylene chloride. Attach the Snyder column to the evaporation flask. Place the labeled K-D apparatus on the water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will chatter but the column will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the labeled K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and labeled K-D from the receiver. Label the receiver with the last 6 digits of the sample number.

14.16 Rinse a two-ball micro-Snyder column with methylene chloride. Add another clean, boiling chip to the receiver and attach the two-ball micro-Snyder column. Place the labeled receiver apparatus on a hot water bath (water bath below boiling) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 0.5 mL, remove the apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column.

14.17 Alternately, the extracts can be concentrated on the nitrogen blow-down unit. Place the labeled receiver on the concentration unit and allow a gentle flow of nitrogen to concentrate the extract to approximately 0.5 mL.

14.18 Adjust the final volume to 1.0 mL with methylene chloride in a 1.0 mL Class “A” volumetric tube. Transfer the extract to an auto-sampler vial, cap with a Teflon lined cap, and label with the sample number and test procedure.

14.19 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the GCMS Semi-volatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.20 Extracts are analyzed using SOPs GCMSSV-001 or GCMSSV-004.

15.0 CALCULATIONS

15.1 Not applicable.

16.0 REPORTING LIMIT

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs GCMSSV-001 and GCMSSV-004 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 3550C

22.2 SW-846 3500C

23.0 METHOD MODIFICATIONS

23.1 Procedure step 14.7 – Sodium sulfate is added to the sample prior to addition of surrogate and spike solutions.

23.2 Procedure step 14.9 -The extraction solvent is added to the sample to be approximately 2 inches above the sediment in the beaker. Exactly 100 mL is not measured as stated in the method.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep sheets are available on GCAL's Intranet under "Prep Menu"

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
		Previous version	
04/11/13	19	New Format. Added several sections for compliance with TNI requirements for SOPs; revised Section 7.0 Safety for clarity; revised Section 9.0 to reflect standard preparation in acetone rather than methanol; revised Section 14.0 to include the nitrogen blow-down unit, and 2 mL vials.	R. Jason Olivier Scott Bailey Shelley Bourgeois
04/11/14	20	Changed Procedure 14.6 to reflect the sample size of 30±0.5 grams. Updated spiking solution for 625 working spike.	Thomas Steiner

STANDARD OPERATING PROCEDURE FOR

ULTRASONIC EXTRACTION FOR PESTICIDES AND PCBs

SW-846 3550C

Approved by: _____ Date: 4/4/14
Thomas Steiner, Extractions Supervisor

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Approved by: _____ Date: 4/4/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This procedure is designed for the preparation of solid samples for the analysis of Organochlorine Pesticides and PCBs

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Soil

3.0 SUMMARY OF TEST METHOD

3.1 A measured volume of sample, approximately 30 g, or a reduced volume based on sample matrix, is serially extracted with 1:1 hexane/acetone using ultrasonic extraction.

3.2 The hexane extract is dried, concentrated, and bottled for analysis of Pesticides and/or PCBs by GC.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware and other sample extraction apparatus may yield interferences to sample analysis. All these must be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

6.4 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Organochlorine pesticides, phthalate esters and phenols may react under basic conditions. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Glass beakers – 250 mL

8.2 Kuderna-Danish (K-D) apparatus

- 8.2.1 Receiver, 10 mL graduated
- 8.2.2 Evaporation Flask, 500 mL - attached to the receiver with a blue clamp
- 8.2.3 3-Ball Snyder Column, pre-rinsed three times with hexane

8.3 2-Ball Snyder Column, pre-rinsed three times with hexane

8.4 Hot plate

8.5 Spatula, stainless steel or wooden stirrer.

8.6 1 mL pipette (verified daily before use) or 1 mL gas tight syringe

8.7 Funnels, glass or Teflon

8.8 Filter Paper – Whatman 41 18.5 cm or equivalent.

8.9 Boiling chips, Teflon or equivalent

8.10 Sonicator- Fisher Scientific Ultrasonic Dismembrator equipped with ¾ inch disruptor horn.

8.11 Pasteur pipets

8.12 Water bath – Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control (± 5°C). The bath must be used in a hood.

8.13 Analytical balance – capable of weighing to 0.1g and calibrated daily when in use

8.14 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps

8.15 Volumetric Flasks – 1.0 mL, 5.0 mL, 10.0 mL

8.16 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps and 20 mL vials with Teflon lined screw caps
8.17 44 mL VOA vials
8.18 Nitrogen Blow-Down Unit

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor.
Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized water
9.1.2 Sodium sulfate (ACS) – granular, anhydrous, heated to approximately 400 °C for a minimum of 4 hours in a shallow tray, cooled and stored in a glass containers with Teflon lined caps. Record in the Sodium Sulfate Logbook and assign a lot number. Label all glass containers of sodium sulfate with the batch number, the date baked, expiration date and the initials of the preparer.
9.1.3 Hexane
9.1.4 Acetone
9.1.5 1:1 Hexane/Acetone

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Surrogate Stock Standards (commercially prepared, Tetrachloro-m-xylene and Decachlorobiphenyl, 200 µg/mL each) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.
9.2.2 Surrogate Working Solution Mix (0.5 µg/mL) – add 2.5 mL of stock Pesticide/PCB Surrogate Mix to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook.

Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.3 608 Stock Spiking Solution (commercially prepared, multi-analyte mix, 1000 µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.4 608 Working Spiking Solution (0.5 µg/mL) – add 250 µL of stock pesticide spiking solution to a 500 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.5 PCB Stock Spiking Solutions (commercially prepared individual spiking solutions of 5000 µg/mL of Aroclor-1016 and 5000 µg/mL of Aroclor-1260) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.6 PCB Working Spiking Solution (4 µg/mL) – add 800 µL of each stock PCB spiking solution to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.7 Commercially prepared standards at the desired concentration or diluted with the appropriate solvent may have to be purchased based on the project or client specified analyte list. These standards will expire according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem. Store standards according to manufacturer's instructions.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 14 days from collection to extraction and 40 days from extraction to analysis for Pesticides; 1 year from collection to extraction and 1 year from extraction to analysis for PCBs.

12.0 QUALITY CONTROL

12.1 One method blank, LCS, LCSD, MS, and MSD shall be prepared per batch, a maximum of twenty samples.

12.2 If insufficient sample is available for a MS and/or MSD then document this on the Extraction Preparation Form.

12.3 QC samples are prepared in the exact manner of the samples using the Procedure in Section 14.0.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified each day of use. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook associated with each pipette.

14.0 PROCEDURE

14.1 Rinse all 250 mL beakers with hexane three times. Dispose of the rinsate in the appropriate solvent waste container.

14.2 Rinse a K-D apparatus three times with hexane. Dispose of the rinsate in the appropriate solvent waste container.

14.3 Label each beaker and K-D apparatus with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.

14.4 With each extraction batch, a method blank, LCS, LCSD and MS/MSD are included. If insufficient sample is available for a MS and/or MSD, document on the Extraction Preparation Form.

14.5 Weigh a 30 g aliquot of sodium sulfate into a hexane rinsed beaker labeled Blank; repeat for the LCS and LCSD. Record the weight on the Extraction Preparation Form.

14.6 Homogenize sample by pouring the contents of the sample container onto a piece of butcher paper and chop and mix with a spatula. Remove any material such as sticks, leaves, or rocks. Weigh 30 ± 0.5 g of homogenized sample to the nearest 0.1 g into the beaker. Record weight on the Extraction Preparation Form.

14.7 Add approximately 30 g of sodium sulfate to each sample and mix well. The sample should have a free flowing sandy texture at this point. If not, add Sodium Sulfate until the sample is free flowing.

14.8 Add 1.0 mL of the surrogate standard spiking solution to each beaker. Spike the LCS/LCSD and the MS/MSD beakers with 1.0 mL of the Pesticide or PCB spiking solutions for samples requiring 8081/8082 analysis. Note: if both pesticides and PCBs are required, the LCS/LCSD and MS/MSD are prepared separately with the associated standards. Mix well. All extraction prep batches require a spike witness that is a peer technician, analyst or someone from supervisory or management level. Only employees that have received spike witness training can act as a spike witness. In the event that an employee is working outside of normal business hours and no qualified spike witness is available, then it is solely the individual technician's responsibility to verify all appropriate spikes and surrogates have been added. In this event, no spike witness will be indicated on the Extraction Preparation Form.

14.9 Immediately add approximately 100 mL of 1:1 hexane/acetone. The solvent level should be about 2 inches above the sample in the beaker.

14.10 The sonicator is tested daily prior to use by pressing the test button on the front panel. If the sonicator is operating correctly the instrument returns to ready mode and is ready for use. If an error message is given, notify supervisor and do not sonicate samples using this sonicator.

14.11 Sonicate each sample and QC samples for 3 minutes by placing the probe of the sonicator about ½ inch below the surface of the solvent but above the sediment.

14.12 Decant and filter extracts through a funnel lined with folded filter paper into a labeled K-D apparatus.

14.13 Repeat the extraction two more times with two additional 100 mL portions of 1:1 hexane/acetone. Combine the solvent from each extraction into the labeled K-D.

14.14 On the final sonication transfer the entire contents of the beaker into the funnel and rinse with hexane.

14.15 Add one or two Teflon boiling chips to the labeled K-D apparatus. Rinse the 3 ball Snyder column with hexane. Attach the Snyder column to the evaporation flask. Place the labeled K-D apparatus on the water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will chatter but the column will not flood with condensed solvent.

14.16 When the apparent volume of liquid reaches approximately 2 mL, remove the labeled K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and labeled K-D from the receiver. Label the receiver with the last 6 digits of the sample number.

14.17 If additional concentration is required, rinse a two-ball micro-Snyder column with hexane. Add another clean, boiling chip to the receiver and attach the two-ball micro-Snyder column. Place the labeled receiver apparatus on a hot water bath (water bath below boiling) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 2 mL, remove the apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column.

14.18 Alternately, the extracts can be concentrated on the nitrogen blow-down unit. Place the labeled receiver on the concentration unit and allow a gentle flow of nitrogen to concentrate the extract to approximately 2 mL.

14.19 Adjust the final volume to 10.0 mL with hexane in a 10.0 mL volumetric flask. Transfer approximately 1 mL of each extract to an auto-sampler vial, cap with a Teflon lined cap, and label with the sample number and test procedure. Discard the remaining extract.

14.20 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the GC Semi-volatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.21 Extracts are analyzed using SOPs GC-012, GC-013, or GC-023.

15.0 CALCULATIONS

15.1 Not applicable.

16.0 REPORTING LIMIT

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs GC-012, GC-013, or GC-023 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 3550C

22.2 SW-846 3500C

23.0 METHOD MODIFICATIONS

23.1 Procedure step 14.7 – Sodium sulfate is added to the sample prior to addition of surrogate and spike solutions.

23.2 Procedure step 14.9 -The extraction solvent is added to the sample to be approximately 2 inches above the sediment in the beaker. Exactly 100 mL is not measured as stated in the method.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep sheets are available on GCAL's Intranet under "Prep Menu"

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
	17	Previous version	
04/11/13	18	New Format. Added several sections for compliance with TNI requirements for SOPs; revised Section 7.0 Safety for clarity; revised Section 9.0 to reflect standard preparation in acetone rather than methanol; revised Section 14.0 to include the nitrogen blow-down unit, and 2 mL vials.	R. Jason Olivier Scott Bailey Shelley Bourgeois
10/08/13	19	Changed extraction solvent from 1:1 methylene chloride/acetone to 1:1 hexane/acetone.	Scott Bailey
04/04/14	20	Changed Procedure 14.6 to reflect the sample size of 30±0.5 grams. Updated Procedure 14.19. The excess extract is discarded.	Scott Bailey Thomas Steiner

STANDARD OPERATING PROCEDURE FOR

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION FOR BASE/NEUTRAL AND ACID COMPOUNDS (BNA)

EPA 3510C / EPA 625

Approved by: _____ Date: 3/26/14
Thomas Steiner, Extractions Supervisor

Approved by: _____ Date: 3/26/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 3/26/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This procedure is designed for the preparation of aqueous samples and TCLP extracts which may contain extractable base, neutral, and acid organic compounds.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water

3.0 SUMMARY OF TEST METHOD

3.1 A measured volume of sample, approximately 1 L, or a reduced volume based on sample matrix, is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel.

3.2 The extract is dried, concentrated, and bottled for analysis of BNAs by GC/MS.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware and other sample extraction apparatus may yield interferences to sample analysis. All these must be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

6.4 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Organochlorine pesticides, phthalate esters and phenols may react under basic conditions. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Separatory Funnel – 2 L Teflon with Teflon stopcock

8.2 Kuderna-Danish (K-D) apparatus

- 8.2.1 Receiver, 10 mL graduated
- 8.2.2 Evaporation Flask, 500 mL - attached to the receiver with a blue clamp
- 8.2.3 3-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.3 2-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.4 Hot plate

8.5 Glass wool, pre-rinsed with methylene chloride

8.6 Glass graduated cylinders – 1000 mL and 100 mL

8.7 Volumetric flasks – 1000 mL and 200 mL

8.8 1 mL pipette (verified daily before use) or 1 mL gas tight syringe

8.9 Funnels, glass or Teflon – packed with glass wool and prepared with sodium sulfate. The funnel and glass wool are rinsed with methylene chloride; collect the rinsate in an Erlenmeyer flask and discard in the appropriate waste container.

8.10 Mechanical shaker

8.11 Boiling chips, Teflon or equivalent

8.12 pH indicator paper with pH range 2-14

8.13 Pasteur pipets

8.14 Glass beakers

8.15 Water bath – Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control (± 5°C). The bath must be used in a hood.

8.16 Analytical balance – capable of weighing to 0.1g and calibrated daily when in use

8.17 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps
8.18 Erlenmeyer flask
8.19 Nitrogen Blow-Down Unit

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor.
Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized water
9.1.2 Sodium hydroxide solution, 50% (ACS) – Dissolve 1000 g NaOH into 1000 mL of DI water. Record reagent preparation in the Standards Preparation Logbook. Label the sodium hydroxide solution container with the standard ID, expiration date, date prepared, and preparer's initials.
9.1.3 Sodium sulfate (ACS) – granular, anhydrous, heated to approximately 400 °C for a minimum of 4 hours in a shallow tray, cooled and stored in a glass containers with Teflon lined caps. Record in the Sodium Sulfate Logbook and assign a lot number. Label all glass containers of sodium sulfate with the batch number, the date baked, expiration date and the initials of the preparer.
9.1.4 Sulfuric acid solution (1:1) – 500 mL H_2SO_4 added slowly to 500 mL reagent water. Record reagent preparation in the Standards Preparation Logbook. Label the sulfuric acid solution container with the standard ID, expiration date, date prepared, and preparer's initials.
9.1.5 Methylene chloride
9.1.6 Acetone

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Surrogate Stock Standards (commercially prepared) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the

manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.1.1 Base/Neutral Surrogate Mix (5000 µg/mL)

9.2.1.2 Acid Surrogate Mix (10,000 µg/mL)

9.2.2 Surrogate Working Solution Mix (50/100 µg/mL) – add 10.0 mL of Base/Neutral Surrogate Mix solution and 10.0 mL of Acid Surrogate Mix solution to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.3 625 Stock Spiking Solution (commercially prepared, multi-analyte mix, 100 µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.4 625 Working Spiking Solution (50 µg/mL) – add 25 mL of 625 stock standard to 25 mL of acetone. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.5 Base Neutral Stock Spiking Solution (commercially prepared, multi-analyte mix, 5000 µg/mL) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.6 Acid Stock Spiking Solution (commercially prepared, multi-analyte mix, 7500 µg/mL) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.7 8270 Working Spiking Solution (100 µg/mL) – add 4.0 mL of Base/Neutral spiking solution and 2.68 mL of Acid spiking solution to a 200 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.8 APP9 Stock Spiking Solution (commercially custom prepared, multi-analyte mix, 400µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.9 APP9 Working Spiking Solution (100 µg/mL) – add 5.0 mL of APP9 stock spiking solution to a 20 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.10 Commercially prepared standards at the desired concentration or diluted with the appropriate solvent may have to be purchased based on the project or client specified analyte list. These standards will expire according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem. Store standards according to manufacturer's instructions.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 7 days from collection to extraction and 40 days from extraction to analysis

12.0 QUALITY CONTROL

12.1 One method blank, LCS, LCSD, MS, and MSD shall be prepared per batch, a maximum of twenty samples. **Note:** an LCSD and MSD are not required for method 625.

12.2 If insufficient sample is available for a MS and/or MSD then document this on the Extraction Preparation Form.

12.3 QC samples are prepared in the exact manner of the samples using the Procedure in Section 14.0.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified each day of use. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook associated with each pipette.

14.0 PROCEDURE

14.1 Rinse all separatory funnels with methylene chloride three times. Dispose of the rinsate in the appropriate solvent waste container.

14.2 Rinse a K-D apparatus three times with methylene chloride. Dispose of the rinsate in the appropriate solvent waste container.

14.3 Label each separatory funnel and K-D apparatus with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.

14.4 With each extraction batch, a method blank, LCS, LCSD and MS/MSD are included. 625 extraction batches only require a method blank, LCS and MS. If insufficient sample is available for a MS and/or MSD, document on the Extraction Preparation Form.

14.5 Measure 1000 mL DI water with a methylene chloride rinsed glass graduated cylinder and transfer into a rinsed separatory funnel labeled Blank; repeat for the LCS and LCSD. Record the volume on the Extraction Preparation Form.

14.6 Use a Sharpie to mark the water meniscus on each sample container. This mark will later be used to determine the initial sample volume.

14.7 Transfer the entire contents of the sample container into a labeled separatory funnel.

14.8 For samples requiring an extraction using a reduced volume (any volume other than the entire contents of the sample container), measure the appropriate sample volume into a 1000 mL graduated cylinder and adjust the sample volume to 1000 mL using DI water. Transfer the contents of the graduated cylinder into a labeled separatory funnel. The volume of sample used will be recorded on the Extraction Preparation Form.

14.9 Add 1.0 mL of the surrogate working solution to each separatory funnel. Spike the LCS/LCSD and the MS/MSD separatory funnels with 1.0 mL of the required 8270 or 625 working spiking solution. If Appendix IX compounds are required, add 1.0 mL of the APP9 spiking solution. Note: if multiple spiking solutions are required, the LCS/LCSD and MS/MSD are prepared separately with the associated standards. Mix well. All extraction prep batches require a spike witness that is a peer technician, analyst or someone from supervisory or management level. Only employees that have received spike witness training can act as a spike witness. In the event that an employee is working outside of normal business hours and no qualified spike witness is available, then it is solely the

individual technician's responsibility to verify all appropriate spikes and surrogates have been added. In this event, no spike witness will be indicated on the Extraction Preparation Form.

14.10 Check the pH of each sample using a Pasteur pipette and strip of pH paper. Adjust the pH of the sample to a pH of < 2 by using 1:1 sulfuric acid. This should take approximately 3 mL for most samples. If the sample requires more than 10 mL of the 1:1 sulfuric acid solution, notify the project manager and note the volume of sulfuric acid required on the Extractions Preparation form.

14.11 Use approximately 60 mL of methylene chloride to rinse the sample bottle. Transfer this rinse solvent to the labeled separatory funnel with the sample. Cap and shake the funnel once then vent to release pressure. Place the capped funnel on the mechanical shaker and set timer for two minutes to extract. Allow the organic (lower) layer to separate from the water phase for at least 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the technician must employ mechanical techniques to effect a phase separation. Optimum techniques depend upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Filter the methylene chloride extract directly through a prepared funnel and collect in a labeled K-D apparatus, fitted with a 10 mL graduated receiver.

NOTE: If significant amounts of sediment are in the bottom of the sample container, it may not be possible to rinse with the solvent. Contact project management to determine additional instructions from the client. If the client requests only the liquid phase be analyzed, the liquid-liquid extraction can be used. See SOP EXT-004. If the client requests both phases analyzed, separate the phases and proceed. Solids are extracted using SOP EXT-001.

14.12 Add a second 60 mL volume of methylene chloride to the labeled separatory funnel and repeat the extraction a second time. Allow the organic layer to separate approximately five minutes and then combine this extract with the extract from section 14.11. Perform a third extraction in the same manner as the second.

14.13 Add approximately 7 mL of the sodium hydroxide solution to each QC sample and sample. Check the pH and verify it is > 11. Additional sodium hydroxide solution is added if the pH is not > 11. Serially extract three times with 60 mL aliquots of methylene chloride in the same manner as the acid extraction was performed. Collect and combine the basic extract and the acid extract into the labeled K-D apparatus.

14.14 Using the methylene chloride rinse bottle, rinse the funnel containing the sodium sulfate into the labeled K-D. This will flush any organics that may have adhered to the sodium sulfate.

14.15 The initial sample volume is determined by filling the sample container with water to the mark indicating the water meniscus. Pour the entire contents into a 1000 mL graduated cylinder. Record the initial volume on the Extraction Preparation Form.

14.16 Add one or two Teflon boiling chips to the labeled K-D apparatus. Rinse the 3 ball Snyder column with methylene chloride. Attach the Snyder column to the evaporation flask. Place the labeled K-D apparatus on the water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will chatter but the column will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the labeled K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and labeled K-D from the receiver. Label the receiver with the last 6 digits of the sample number.

14.17 Rinse a two-ball micro-Snyder column with methylene chloride. Add another clean, boiling chip to the receiver and attach the two-ball micro-Snyder column. Place the labeled receiver apparatus on a hot water bath (water bath below boiling) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 0.5 mL, remove the apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column.

14.18 Alternately, the extracts can be concentrated on the nitrogen blow-down unit. Place the labeled receiver on the concentration unit and allow a gentle flow of nitrogen to concentrate the extract to approximately 0.5 mL.

14.19 Adjust the final volume to 1.0 mL with methylene chloride in a 1.0 mL Class “A” volumetric tube. Transfer the extract to an auto-sampler vial, cap with a Teflon lined cap, and label with the sample number and test procedure.

14.20 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the GCMS Semi-volatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.21 Extracts are analyzed using SOPs GCMSSV-001, GCMSSV-002, or GCMSSV-004.

15.0 CALCULATIONS

15.1 Not applicable.

16.0 REPORTING LIMIT

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs GCMSSV-001, GCMSSV-002, and GCMSSV-004 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 3510C

22.2 EPA 3500C

22.3 EPA 625

23.0 METHOD MODIFICATIONS

The following are modifications of EPA 625 to correspond with SW-846 Method 3510C guidance:

23.1 The acid extraction is performed first. This is done to improve the recovery of phenols that can decompose if the sample is made basic prior to the acid extraction.

23.2 The base-neutral and acid extracts are combined prior to concentration.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep sheets are available on GCAL's Intranet under "Prep Menu"

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
		Previous version	
11/26/12	22	New Format. Added Section 5.0 Interferences; revised Section 9.0 Safety for clarity; revised Section 14.0 to include the nitrogen blow-down unit, methylene chloride rinsed glass wool and 2 mL vials; revised Section 11, 12 and 13 for clarity; added the glossary and standards prep logbook.	R. Jason Olivier Scott Bailey
04/01/13	23	Added method reference for EPA 625; standard preparation in acetone rather than methanol; format updates.	Shelley Bourgeois
03/26/14	24	Updated spiking solution for 625 working spike.	Thomas Steiner

STANDARD OPERATING PROCEDURE FOR

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION FOR PESTICIDES AND PCBs

SW-846 3510C
EPA 608

Approved by: _____ Date: 01/10/14
Thomas Steiner, Extractions Supervisor

Approved by: _____ Date: 01/10/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 01/10/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This procedure is designed for the preparation of aqueous samples and TCLP extracts for analysis of Organochlorine Pesticides and PCBs.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water

3.0 SUMMARY OF TEST METHOD

3.1 A measured volume of sample, approximately 1 L, or a reduced volume based on sample matrix, is serially extracted with methylene chloride at a pH between 5 and 9 using a separatory funnel.

3.2 The methylene chloride extract is dried, exchanged to hexane, concentrated, and bottled for analysis of Pesticides and/or PCBs by GC.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware and other sample extraction apparatus may yield interferences to sample analysis. All these must be demonstrated to be free from interferences under the conditions of analysis by analyzing method blanks.

6.2 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

6.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

6.4 Soap residue, which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Organochlorine pesticides, phthalate esters and phenols may react under basic conditions. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Separatory Funnel – 2 L Teflon with Teflon stopcock

8.2 Kuderna-Danish (K-D) apparatus

- 8.2.1 Receiver, 10 mL graduated
- 8.2.2 Evaporation Flask, 500 mL - attached to the receiver with a blue clamp
- 8.2.3 3-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.3 2-Ball Snyder Column, pre-rinsed three times with methylene chloride

8.4 Hot plate

8.5 Glass wool, pre-rinsed with methylene chloride

8.6 Glass graduated cylinders – 1000 mL and 100 mL

8.7 Volumetric flasks – 1000 mL, 100 mL, and 500 mL

8.8 1 mL pipette (verified daily before use) or 1 mL gas tight syringe

8.9 Funnels, glass or Teflon – packed with glass wool and prepared with sodium sulfate. The funnel and glass wool are rinsed with methylene chloride; collect the rinsate in an Erlenmeyer flask and discard in the appropriate waste container.

8.10 Mechanical shaker

8.11 Boiling chips, Teflon or equivalent

8.12 pH indicator paper with pH range 2-14

8.13 Pasteur pipets

8.14 Glass beakers

8.15 Water bath – Organomation S-Evap-KD Solvent Evaporator, or equivalent, heated with concentric ring cover and capable of temperature control (± 5 °C). The bath must be used in a hood.

8.16 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps and 20 mL vials with Teflon lined screw caps
8.17 44 mL VOA vials
8.18 Erlenmeyer flask
8.19 Nitrogen Blow-Down Unit

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor.
Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized water
9.1.2 Sodium hydroxide solution, 50% (ACS) – Dissolve 1000 g NaOH into 1000 mL of DI water. Record reagent preparation in the Standards Preparation Logbook. Label the sodium hydroxide solution container with the standard ID, expiration date, date prepared, and preparer's initials.
9.1.3 Sodium sulfate (ACS) – granular, anhydrous, heated to approximately 400 °C for a minimum of 4 hours in a shallow tray, cooled and stored in a glass containers with Teflon lined caps. Record in the Sodium Sulfate Logbook and assign a lot number. Label all glass containers of sodium sulfate with the batch number, the date baked, expiration date and the initials of the preparer.
9.1.4 Sulfuric acid solution (1:1) – 500 mL H_2SO_4 added slowly to 500 mL reagent water. Record reagent preparation in the Standards Preparation Logbook. Label the sulfuric acid solution container with the standard ID, expiration date, date prepared, and preparer's initials.
9.1.5 Methylene chloride
9.1.6 Acetone
9.1.7 Hexane

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all

standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Surrogate Stock Standards (commercially prepared, Tetrachloro-m-xylene and Decachlorobiphenyl, 200 µg/mL each) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.2 Surrogate Working Solution Mix (0.5 µg/mL) – add 2.5 mL of stock Pesticide/PCB Surrogate Mix to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.3 608 Stock Spiking Solution (commercially prepared, multi-analyte mix, 1000 µg/mL) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.4 608 Working Spiking Solution (0.5 µg/mL) – add 250 µL of stock pesticide spiking solution to a 500 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.5 PCB Stock Spiking Solutions (commercially prepared individual spiking solutions of 5000 µg/mL of Aroclor-1016 and 5000 µg/mL of Aroclor-1260) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.6 PCB Working Spiking Solution (4 µg/mL) – add 800 µL of each stock PCB spiking solution to a 1000 mL volumetric flask and bring to volume with acetone. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.7 Commercially prepared standards at the desired concentration or diluted with the appropriate solvent may have to be purchased based on the

project or client specified analyte list. These standards will expire according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem. Store standards according to manufacturer's instructions.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 7 days from collection to extraction and 40 days from extraction to analysis for Pesticides; 1 year from collection to extraction and 1 year from extraction to analysis for PCBs.

12.0 QUALITY CONTROL

12.1 One method blank, LCS, LCSD, MS, and MSD shall be prepared per batch, a maximum of twenty samples. **Note:** an LCSD and MSD are not required for method 608.

12.2 If insufficient sample is available for a MS and/or MSD then document this on the Extraction Preparation Form.

12.3 QC samples are prepared in the exact manner of the samples using the Procedure in Section 14.0.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified each day of use. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook associated with each pipette.

14.0 PROCEDURE

14.1 Rinse all separatory funnels with methylene chloride three times. Dispose of the rinsate in the appropriate solvent waste container.

14.2 Rinse a K-D apparatus three times with methylene chloride. Dispose of the rinsate in the appropriate solvent waste container.

14.3 Label each separatory funnel and K-D apparatus with the last six (6) digits of the sample ID to be extracted. Fill in the Extraction Preparation Form information as each item is completed.

14.4 With each extraction batch, a method blank, LCS, LCSD and MS/MSD are included. 608 extraction batches only require a method blank, LCS and MS. If insufficient sample is available for a MS and/or MSD, document on the Extraction Preparation Form.

14.5 Measure 1000 mL DI water with a methylene chloride rinsed glass graduated cylinder and transfer into a rinsed separatory funnel labeled Blank; repeat for the LCS and LCSD. Record the volume on the Extraction Preparation Form.

14.6 Use a Sharpie to mark the water meniscus on each sample container. This mark will later be used to determine the initial sample volume.

14.7 Transfer the entire contents of the sample container into a labeled separatory funnel. If the sample contains a large amount of sediment or an obvious matrix issue would prevent the use of the entire sample container, follow Procedure 14.8 for measuring the sample volume.

14.8 For samples requiring an extraction using a reduced volume (any volume other than the entire contents of the sample container), measure the appropriate sample volume into a 1000 mL graduated cylinder and adjust the sample volume to 1000 mL using DI water. Transfer the contents of the graduated cylinder into a labeled separatory funnel. The volume of sample used will be recorded on the Extraction Preparation Form.

14.9 Add 1.0 mL of the surrogate working solution to each separatory funnel. Spike the LCS/LCSD and the MS/MSD separatory funnels with 1.0 mL of the Pesticide or PCB spiking solutions for samples requiring 8081/8082 or 608 analysis. Note: if both pesticides and PCBs are required, the LCS/LCSD and MS/MSD are prepared separately with the associated standards. Mix well. All extraction prep batches require a spike witness that is a peer technician, analyst or someone from supervisory or management level. Only employees that have received spike witness training can act as a spike witness. In the event that an employee is working outside of normal business hours and no qualified spike witness is available, then it is solely the individual technician's responsibility to verify all appropriate spikes and surrogates have been added. In this event, no spike witness will be indicated on the Extraction Preparation Form.

14.10 Check the pH of each sample using a Pasteur pipette and strip of pH paper. Adjust the pH of the sample to a pH between 5 and 9 by using a sodium hydroxide and/or sulfuric acid solution.

14.11 Use approximately 60 mL of methylene chloride to rinse the sample bottle. Transfer this rinse solvent to the labeled separatory funnel with the sample. Cap and shake the funnel once then vent to release pressure. Place the capped funnel on the mechanical shaker and set timer for two minutes to extract. Allow the

organic (lower) layer to separate from the water phase for at least 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the technician must employ mechanical techniques to effect a phase separation. Optimum techniques depend upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Filter the methylene chloride extract directly through a prepared funnel and collect in a labeled K-D apparatus, fitted with a 10 mL graduated receiver.

NOTE: If significant amounts of sediment are in the bottom of the sample container, it may not be possible to rinse with the solvent. Contact project management to determine additional instructions from the client. If the client requests only the liquid phase be analyzed, the liquid-liquid extraction can be used. See SOP EXT-011. If the client requests both phases analyzed, separate the phases and proceed. Solids are extracted using SOP EXT-002.

14.12 Add a second 60 mL volume of methylene chloride to the labeled separatory funnel and repeat the extraction a second time. Allow the organic layer to separate approximately five minutes and then combine this extract with the extract from section 14.11. Perform a third extraction in the same manner as the second.

14.13 Using the methylene chloride rinse bottle, rinse the funnel containing the sodium sulfate into the labeled K-D. This will flush any organics that may have adhered to the sodium sulfate.

14.14 The initial sample volume is determined by filling the sample container with water to the mark indicating the water meniscus. Pour the entire contents into a 1000 mL graduated cylinder. Record the initial volume on the Extraction Preparation Form.

14.15 Add one or two Teflon boiling chips to the labeled K-D apparatus. Rinse the 3 ball Snyder column with methylene chloride. Attach the Snyder column to the evaporation flask. Place the labeled K-D apparatus on the water bath. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation the balls of the column will chatter but the column will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, add approximately 44 mL of Pesticide-grade hexane through the Snyder column.

14.16 Continue the distillation described in Procedure 14.15. When the apparent volume of liquid reaches approximately 2 mL, remove the labeled K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and labeled K-D from the receiver. Label the receiver with the last 6 digits of the sample number.

14.17 If additional concentration is required, rinse a two-ball micro-Snyder column with

hexane. Add another clean, boiling chip to the receiver and attach the two-ball micro-Snyder column. Place the labeled receiver apparatus on a hot water bath (water bath below boiling) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 2 mL, remove the apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column.

14.18 Alternately, the extracts can be concentrated on the nitrogen blow-down unit. Place the labeled receiver on the concentration unit and allow a gentle flow of nitrogen to concentrate the extract to approximately 2 mL.

14.19 Adjust the final volume to 10.0 mL with hexane in a 10.0 mL volumetric flask for SW846 8081/8082 and to 5.0 mL with hexane using a 5.0 mL volumetric flask for EPA 608 samples. Transfer approximately 1 mL of each extract to an auto-sampler vial, cap with a Teflon lined cap, and label with the sample number and test procedure. Discard the remaining extract.

14.20 Sign the Extraction Preparation Form. Deliver a copy of the Extraction Preparation Form and the extracts to the GC Semi-volatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator with the Extraction Preparation Form.

14.21 Extracts are analyzed using SOPs GC-012, GC-013, or GC-023.

15.0 CALCULATIONS

15.1 Not applicable.

16.0 REPORTING LIMIT

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs GC-012, GC-013, and GC-023 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 3510C

22.2 SW-846 3500C

22.3 EPA 608

23.0 METHOD MODIFICATIONS

23.1 Method 3510C states that 50 mL of exchange solvent should be used. GCAL's procedure is to use approximately 44 mL (the contents of a full VOA vial).

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep sheets are available on GCAL's Intranet under "Prep Menu"

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
		Previous version	
04/01/13	15	New Format. Added Section 6.0 Interferences; revised Section 7.0 Safety for clarity; revised Section 9.0 to reflect standard preparation in acetone rather than methanol; revised Section 14.0 to include the nitrogen blow-down unit, methylene chloride rinsed glass wool and 2 mL vials. Added method reference for EPA 608.	R. Jason Olivier Scott Bailey Shelley Bourgeois
01/10/14	16	Clarified Procedure 14.7 on appropriate situations for preparation of reduced volume. Updated the language in 14.9 to reflect spiking 1.0 mL for all samples (Previously 1.0 mL for 8081B and 0.5 mL for 608). Updated 14.19 – excess sample extract is no longer stored.	Shelley Bourgeois Thomas Steiner

STANDARD OPERATING PROCEDURE FOR

ORGANOCHLORINE PESTICIDES AND PCBs BY GAS CHROMATOGRAPHY

EPA 608 / EPA 8081B / EPA 8082A

Approved by: _____ Date: 4/11/14
Donna Broussard, Semivolatiles Supervisor

Approved by: _____ Date: 4/11/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 4/11/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This method provides gas chromatographic procedures for the detection of ppb levels of certain Organochlorine pesticides and PCBs. A list of target analytes is included in Table 1.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water, Soil, and Waste

3.0 SUMMARY OF TEST METHOD

3.1 Prior to the use of this method, appropriate extraction techniques must be used. Pesticides are extracted using SOP EXT-002, EXT-010, EXT-011, or EXT-037. An aliquot of the sample extract is injected into a GC equipped with an ECD detector.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware, and other sample artifacts may interfere with sample analysis. Interferences are monitored by the analysis of a method blank performed with each batch.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when

working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

7.6 The electron capture detectors employed in this method must have a wipe test performed twice a year. In accordance with the federally mandated, state administered programs, the wipes are checked for degradation of the radionuclide foil by a contracted nuclear counting firm. Evidence of this test and its results are kept on file at GCAL.

8.0 EQUIPMENT AND SUPPLIES

8.1 Gas Chromatograph should be suitable for split-less or on-column injection. The system should be equipped with an Electron Capture Detector. The instrument used is an Agilent GC 6890N. Recommended GC Operating conditions:.

8.1.1 Oven Program – Set initial temperature to 120°C, ramp at 45°C per minute to 200°C, then ramp 15°C per minute to 230°C, then ramp 30°C per minute to 330°C, hold for 1 minute.

8.1.2 Sample/auto-sampler injection volume – 1 µL

8.1.3 Carrier Gas – Hydrogen

8.1.4 ECD Temperature – 350°C

8.1.5 Injection Port Temperature – 250°C

8.2 The Agilent GC utilizes Agilent software GC Chemstation Version A.10.02 to control the instrument and transfer data acquisition to Target Data Integration software. Target software is utilized to calculate all data. In the event of software malfunction for the GC Chemstation, please contact Agilent technical support for troubleshooting help.

8.3 The data system must be capable of time stamping all data produced with the correct date and time. The data system employed will be Target. In the event of software malfunction when using Target, please contact GCAL's IT Director for troubleshooting help.

8.4 XLB or similar phase 30m X 0.32mm ID

8.5 35MS or similar phase 30m X 0.32mm ID

8.6 50 mL volumetric flask with stopper

8.7 2 mL auto-sampler vials with Teflon lined crimp caps or screw caps

8.8 Gas-tight syringe

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Pesticide quality and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the

expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Hexane, pesticide grade or equivalent

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock Standards

9.2.1.1 Surrogate Stock Standards (commercially prepared, Tetrachloro-m-xylene and Decachlorobiphenyl, 200 µg/mL each) – store refrigerated at ≤ 6 °C in sealed ampules until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.1.2 Pesticide Stock Spiking Solution (commercially prepared, multi-analyte mix, varied concentrations) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.1.3 Toxaphene (commercially prepared, 5000 µg/mL)

9.2.1.4 Chlordane, technical (commercially prepared, 1000 µg/mL)

9.2.1.5 Single analyte compounds (Alachlor, Chlorobenzene, Diallate, Isodrin, Kepone, Mirex) – purchase as needed

9.2.1.6 PCB Stock Spiking Solutions (commercially prepared, Aroclor-1016/1260, 5000 µg/mL each) – store refrigerated at ≤ 6 °C in amber glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.1.7 Aroclors (commercially prepared, Aroclor 1221, 1232, 1242, 1248, 1254, 1262, and 1268, 100ug/ml)

9.2.1.8 Degradation (DEG) Stock Standard (commercially prepared, 4,4'-DDT and Endrin, 200 µg/mL each)

9.2.2 Working Standards – store refrigerated at ≤ 6 °C in a glass bottle until use. Expires according to the manufacturer's expiration date or 6 months after preparation whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.2.1 Pesticide Working Spiking Solution (varied concentrations) – add 625 µL of stock pesticide spiking solution and 25 µL of the surrogate solution to a 50 mL volumetric flask and bring to volume with hexane. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.2.2 Toxaphene Working Standard – add 50 µL of stock toxaphene spiking solution to a 50 mL volumetric flask and bring to volume with hexane. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.2.3 Chlordane Working Standard – add 20 µL of stock toxaphene spiking solution to a 50 mL volumetric flask and bring to volume with hexane. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.2.4 PCB Working Spiking Solution (4 µg/mL) – For AR 1221, 1232, 1242, 1248 and 1254 add 200 µL of each stock PCB to a 50 mL volumetric flask and bring to volume with hexane. For AR 1262 add 20 µL of PCB spiking solution to a 50 mL volumetric flask and bring to volume with hexane. For AR 1268 add 40 µL of PCB spiking solution to a 50 mL volumetric flask and bring to volume with hexane. Record in the Standards Preparation Logbook. Transfer into an amber glass bottle with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store refrigerated at ≤ 6 °C until use.

9.2.3 Calibration Standards – The calibration standards are prepared by diluting a stock solution into 3 to 7 working levels. Table 2 indicates the Aroclor concentration. The standard is diluted with Hexane.

9.2.3.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 is diluted into 6 working levels. The remaining five Aroclors (1221, 1232, 1242, 1248, and 1254), Technical Chlordane and Toxaphene are calibrated using one working level of each. Toxaphene is at 5000 ug/L. All others are at 400ug/L.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen, protect from light

11.2 Maximum holding time - Pesticides

11.2.1 Waters – 7 days from collection to extraction and 40 days from extraction to analysis

11.2.2 Solids – 14 days from collection to extraction and 40 days from extraction to analysis

11.3 Maximum holding time - PCBs – 1 year from collection to extraction and 1 year from extraction to analysis

12.0 QUALITY CONTROL

12.1 One method blank shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One LCS and/or LCS/LCSD shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to DI water or sodium sulfate and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One MS shall be analyzed per batch, a maximum of twenty samples. The MS is a known amount of standard added to sample at the same concentration as the LCS.

12.4 One matrix spike duplicate (MSD) shall be analyzed per batch, a maximum of 20 samples.

12.5 A surrogate is added to all QC and samples. The purpose of surrogates is to verify that each sample was properly extracted. A surrogate is a non-target

compound that is chemically similar to the analytes. The surrogates used are Tetrachloro-m-xylene and Decachlorobiphenyl. Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Retention Time Windows

13.1.1 For all analytes and surrogates a retention time study is performed over approximately 72 hours whenever a new column is installed, after major maintenance, or during initial set-up. The standard deviation (SD) is calculated based on this study using at least three determinations, measured to 0.001 minutes. The width of the retention time window is three times the SD for each of the analytes. Alternatively, if the calculated SD is less than 0.01 minutes, a default window of ± 0.03 minutes shall be employed.

13.1.2 The daily retention time window for the analyte is equal to the retention time of the analyte in the first CCV of the day, ± 3 times the standard deviation calculated from the retention time study or ± 0.03 minutes when the default is employed.

13.2 Degradation Standard (DEG)

13.2.1 A degradation check standard must be analyzed each day prior to analyzing 8081 and 608 samples and once every 12-hours the instrument continues to analyze samples. Inject 1 µL of the 80 ppb DEG working standard.

13.2.2 The degradation standard contains 4,4'-DDT and Endrin and is used to determine their breakdown components. 4,4'-DDT breaks down to 4,4'-DDD and DDE, Endrin breaks down to Endrin Aldehyde and Endrin Ketone.

13.2.3 The breakdown standard insures the instrument is maintained properly and will not cause breakdown of DDT and Endrin.

13.2.4 The degradation of either 4,4-DDT or Endrin cannot exceed 15% individually.

13.2.5 If the degradation standard fails to meet this criteria take appropriate corrective action (clean injector, replace insert, septa, ferrules, trim column, re-prep. standard, clean syringe, etc.). The degradation standard must meet criteria prior to analyzing samples for Endrin, DDT and their breakdown components.

13.3 Initial Calibration (ICAL) – an ICAL is performed using a blank and 3 to 7 levels of standards at the concentrations noted in Table 1. An initial calibration curve for each single component target analyte must be analyzed and evaluated before any peak for that analyte can be quantitated. Two low-level standards have been added to meet permit limits, but they are only added to the calibration when permit samples or LA RECAP samples are to be analyzed by method 608. For multi-component analytes, Technical Chlordane and Toxaphene, a single point is

analyzed for the ICAL. In the analysis of Aroclors, the calibration is established for a mixture of 1016/1260. This combined mixture demonstrates linearity of the early and late eluting Aroclors. For all other Aroclors a mid-level standard is analyzed. Because six calibration standards are used, the analyst has the option of eliminating one of the points using the criteria below. Within the following requirements the analyst may select the points that improve linearity, obtain the calibration range needed for a specific project, or maintain the default calibration range (5.0 – 100 µg/mL). Typically, alpha-BHC is the only compound reported in the Level 1 standard. For DOD projects and other projects that do not require lower detection limits, levels 2 through 6 are used on a regular basis and alpha-BHC is left on in level 1 at all times for RECAP projects.

13.3.1 Average Response Factor Calibration - Pesticides

13.3.1.1 For each of the standards, calculate the response factor using the equation found in Section 15, Calculations.

13.3.1.2 Method 608 - Calculate the average of the three to five response factors and the standard deviation across the three to five response factors. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD). When the response factors of the standards demonstrate less than 10 %RSD of all target analytes, linearity through the origin can be assumed. If the %RSD is greater than 10% for any analyte of interest, the initial calibration must be re-analyzed.

13.3.1.3 Method 8081B - Calculate the average of a minimum of five response factors and the standard deviation across the selected five response factors. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD). When the five (or more) response factors of the standards demonstrate less than 20% RSD for a target analytes, linearity through the origin can be assumed. If the RSD for any analyte is greater than 20%, the analyst may wish to review the results for those analytes to ensure that the problem is not associated with just one of the initial calibration standards.

13.3.2 Average Response Factor Calibration – PCBs

13.3.2.1 For each of the six levels for Aroclor 1016/1260, calculate the response factor of 5 major characteristic Aroclor peaks in each of the initial calibration standards. It is impossible to choose 5 discreet peaks for each of the nine PCB's because many peaks are the same in several peaks. Because so many peaks are shared between the PCBs, five discreet peaks are chosen for each of the nine PCB's. These five peaks are identified in the chromatograms attached. The five peaks have been chosen distinctly for both columns so each PCB has two patterns attached - one for each column. This was done to provide continuity and repeatability

between varying lab analysts and personnel. Each peak chosen should have a sufficient response to allow for accurate quantitation of the detected Aroclors. The analyst should be aware of the effect of weathering on Aroclor patterns and careful consideration should be given to patterns detected in samples when choosing peaks. Although five peaks are used for quantification, all peaks in the pattern are used in identification. Additionally, a DDT degraded standard run when distinct PCB patterns were chosen. No peak was chosen for a pattern that had the same retention time for DDT, DDD, or DDE to prevent these commonly found environmental constituents from interfering with the quantitative measurements.

13.3.2.2 For Aroclor 1016 and 1260, calculate the average of these six response factors and the standard deviation across the six response factors. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD). When the six response factors of the standards demonstrate less than 20 %RSD for all target analytes, linearity through the origin can be assumed.

13.3.2.3 For each of the single-point midlevel of the remaining Aroclors, calculate the response factor for five of the characteristic Aroclor peak in each single-point.

13.3.3 For those compounds that the RSD exceeds the criteria for Average RF, a linear regression equation that is not forced through the origin may be used. The coefficient of determination must be at least 0.990 for the curve to be acceptable. As part of the evaluation, check the y-intercept (b) and any negative factors (b or M). Negative factors may result in false negatives and positives. Large shifts in the y-intercept from zero will impact the data. If the intercept is greater than half the reporting limit, this option shall not be used. Check the required reporting limit and any impacts on the data. False positives shall be indicated in the case narrative.

13.3.4 A quadratic curve fit may be used if the coefficient of determination $r^2 \geq 0.990$. A minimum of a six-point calibration is used if this option is chosen and the curve shall not be forced through zero. As part of the evaluation, check the y-intercept (b) and any negative factors (b or M). Negative factors may result in false negatives and positives. Large shifts in the y-intercept from zero will impact the data. If the intercept is greater than half the reporting limit, this option shall not be used. Check the required reporting limit and any impacts on the data. False positives shall be indicated in the case narrative. For South Carolina projects, quadratic curve fits are not allowed.

13.3.5 If a compound fails the initial calibration criteria, re-analyze for the compound that failed. If the compound fails in the re-analysis, perform

column maintenance and clean injection port, then re-analyze the initial calibration for all analytes.

13.4 Independent Calibration Verification (ICV) – immediately following the ICAL analyze a standard containing all single component target analytes prepared from a standard that is independent from the ICAL standard. It is preferred that the ICV be from a different manufacturer. If this is not available than a different lot from the same manufacturer may be used.

13.4.1 The ICV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 15% difference from the initial calibration.

13.4.2 If the criterion referenced above is not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved.

13.5 Continuing Calibration Verification (CCV) – each day of analysis that an ICAL is not performed a CCV must be analyzed before sample analysis. The CCV is also analyzed every 10 samples or 12 hours and at the end of the analytical batch.

13.5.1 The CCV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 20% difference from the initial calibration.

13.5.2 If the calibration verification standard fails to meet the criteria, repeat the injection of the standard. If the standard fails again, take appropriate corrective action (inspection of GC, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.

13.5.3 If the calibration verification standard analyzed after a group of samples has a response for an analyte that is high and the analyte was not detected in any of the previous samples during the analytical shift, then the samples do not need to be re-analyzed.

14.0 PROCEDURE

14.1 Sample Analysis

14.1.1 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with a degradation standard followed by an initial calibration or calibration verification. It contains a CCV every 10 injections or 12 hours and ends when the samples in the batch have been analyzed. The analytical sequence is closed with a CCV.

14.1.2 All samples are evaluated on a primary column. If an analyte is detected above the detection limit, then the secondary column is evaluated to confirm the presence of that analyte. If the secondary column is used for confirmation, all ICAL and CCV acceptance criteria must be met on both columns.

14.1.3 If the analyte falls within the retention time window on both the primary and secondary analysis, then calculate the concentration present. The higher value of the two analyses is reported. Specific project requirements

may define how to report analytical results, refer to SOP GEN-019 for further instruction. If the difference between the primary result and the secondary result has a calculated RPD greater than 40%, the sample result must be narrated and the lower result of the two is reported.

14.1.4 If samples have analytes that are above the calibration range, the sample must be diluted. A portion of the sample, measured with a gas tight syringe, is diluted with hexane. The dilution factor is recorded in Target and used to calculate the analyte concentrations and to adjust reporting limits.

14.2 Manual Integrations

14.2.1 See SOP QA-010 for GCAL's Manual Integration Policy.

15.0 CALCULATIONS

15.1 % Recovery (MS/MSD) = $\frac{\text{Spike Concentration-Sample Concentration}}{\text{Spike Added}} \times 100$

15.2 % Recovery (LCS/LCSD/Surrogate) = $\frac{\text{Spike Concentration}}{\text{Spike Added}} \times 100$

15.3 RPD = $\frac{|\text{Concentration 2-Concentration 1}|}{(\text{Concentration 2+Concentration 1})/2} \times 100$

15.4 Response Factor $= A_s/C_s$

As= Peak Area of analyte or surrogate

Cs= Concentration of the analyte or surrogate

15.5 % Difference = $[(RF_I - RF_C) / RF_I] \times 100$

RF_I = Average response factor from initial calibration

RF_C = Response factor from current verification check standard

15.6 % RSD = (SD/X) X 100

RSD = Relative Standard Deviation

X = mean of initial RF's for a compound

SD = Standard Deviation of average RF's for a compound

15.7 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMITS

16.1 See Table 1 for Limit of Quantitation (LOQ) values

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in the analytical methods.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 20 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 10%. This shall be evaluated quarterly. Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the on-column amount measured in any sample (whichever is greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code “RP” for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately.

19.2 The LCS control limits specified in Table 3 are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria, the source of inaccuracy should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code “RP” for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.3 The MS is evaluated against the LCS control limits and is used to determine sample matrix effect. If the MS recovery is outside of control limits and the LCS is within control limits, matrix interference is indicated. Report the results with a case narrative.

19.4 The LCS/LCSD and MS/MSD RPD criteria are specified in Table 3. If the RPD is outside the control limits, investigate the source of the precision error. A source of precision error in the MS/MSD may be the heterogeneous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.5 The surrogate criteria are specified in Table 4. If a surrogate recovery is outside QC limits in a MB, LCS, or LCSD, the entire batch must be re-extracted and/or reanalyzed. Enter condition code "RP" for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If a surrogate recovery is outside QC limits in a sample, and no obvious matrix interference is present, the sample should be re-extracted and/or reanalyzed. If the surrogate recovery is confirmed report the original data prep and analysis. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If obvious chromatographic interference with surrogate is present, reanalysis is not necessary. Report the data with a case narrative. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.6 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.7 Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative.

19.8 Spikes may be diluted out in the analysis process if a 10X or higher dilution is performed. Document this in the case narrative.

19.9 Project specific criteria may apply. See SOP GEN-019.

19.10 For data review procedures and checklists, see SOP QA-002.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 608
22.2 EPA 8000B
22.3 EPA 8081B
22.4 EPA 8082A

23.0 METHOD MODIFICATIONS

23.1 The carrier gas used is Hydrogen rather than Helium

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history
24.2 Table 1: Target Analytes / Limit of Quantitation
24.3 Table 2: Calibration concentrations
24.4 Table 3: Control limits for LCS
24.5 Table 4: Control limits for Surrogate
24.6 Table 5: Criteria for Marginal Sporadic Failures

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
04/10/13	09	Previous version	
04/11/14	10	Updated format and added sections required for TNI-compliant SOPs. Corrected typos. Added tables. Updated control limits. Incorporated SOPs for pesticides and PCBs by methods 608, 8081, and 8082, previously SOPs GC-012, GC-013, and GC-023, into one SOP, GC-012.	Shelley Bourgeois

Table 1: Target Analytes / Limit of Quantitation

Analyte	LOQ 608 Water (µg/L)	LOQ 8081/8082 Water (µg/L)	LOQ 8081/8082 Soil (µg/Kg)
4,4'-DDD	0.050	0.050	3.33
4,4'-DDE	0.050	0.050	3.33
4,4'-DDT	0.050	0.050	3.33
Alachlor	0.050	0.050	3.33
Aldrin	0.025	0.025	1.67
alpha-BHC	0.025	0.025	1.67
alpha-Chlordane	0.025	0.025	1.67
Aroclor-1016 (PCB-1016)	0.5	0.5	33.3
Aroclor-1221 (PCB-1221)	0.5	0.5	33.3
Aroclor-1232 (PCB-1232)	0.5	0.5	33.3
Aroclor-1242 (PCB-1242)	0.5	0.5	33.3
Aroclor-1248 (PCB-1248)	0.5	0.5	33.3
Aroclor-1254 (PCB-1254)	0.5	0.5	33.3
Aroclor-1260 (PCB-1260)	0.5	0.5	33.3
Aroclor-1262 (PCB-1262)	0.5	0.5	33.3
Aroclor-1268 (PCB-1268)	0.5	0.5	33.3
beta-BHC	0.025	0.025	1.67
Chlordane (tech.)	0.25	0.25	16.7
delta-BHC	0.025	0.025	1.67
Diallate	0.050	0.050	3.33
Dieldrin	0.050	0.050	3.33
Endosulfan I	0.025	0.025	1.67
Endosulfan II	0.050	0.050	3.33
Endosulfan sulfate	0.050	0.050	3.33
Endrin	0.050	0.050	3.33
Endrin aldehyde	0.050	0.050	3.33
Endrin ketone	0.050	0.050	3.33
gamma-BHC (Lindane)	0.025	0.025	1.67
gamma-Chlordane	0.025	0.025	1.67
Heptachlor	0.025	0.025	1.67
Heptachlor epoxide	0.025	0.025	1.67
Isodrin	0.025	0.025	3.33

Kepone	0.025	0.025	3.33
Methoxychlor	0.25	0.25	16.7
Mirex	0.025	0.025	3.33
Toxaphene	0.25	0.25	16.7

Table 2A: Pesticide Calibration Concentration (µg/L)

	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6	LEVEL 7
4,4'-DDD	2	5	10	40	120	160	200
4,4'-DDE	2	5	10	40	120	160	200
4,4'-DDT	2	5	10	40	120	160	200
Alachlor	1	2.5	5	20	60	80	100
Aldrin	1	2.5	5	20	60	80	100
alpha-BHC	1	2.5	5	20	60	80	100
alpha-Chlordane	1	2.5	5	20	60	80	100
beta-BHC	1	2.5	5	20	60	80	100
Chlordane (tech.)	x	x	x	x	x	500	x
delta-BHC	1	2.5	5	20	60	80	100
Diallate	x	X	10	40	120	160	200
Dieldrin	2	5	10	40	120	160	200
Endosulfan I	1	2.5	5	20	60	80	100
Endosulfan II	2	5	10	40	120	160	200
Endosulfan sulfate	2	5	10	40	120	160	200
Endrin	2	5	10	40	120	160	200
Endrin aldehyde	2	5	10	40	120	160	200
Endrin ketone	2	5	10	40	120	160	200
gamma-BHC (Lindane)	1	2.5	5	20	60	80	100
gamma-Chlordane	1	2.5	5	20	60	80	100
Heptachlor	1	2.5	5	20	60	80	100
Heptachlor epoxide	1	2.5	5	20	60	80	100
Isodrin	x	X	10	40	120	160	200
Kepone	x	X	10	40	120	160	200
Methoxychlor	10	25	50	200	400	600	1000
Mirex	x	x	10	40	120	160	200
Toxaphene	x	x	x	x	x	5000	x

Table 2B: PCB Calibration Concentration (µg/mL)

	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6	LEVEL 7
Aroclors 1016/1260	20	50	100	200	400	800	1600

Table 3: Control Limits for LCS/LCSD/MS/MSD

Analyte	Control Limits 608 Water (%)	Control Limits 8081/8082 Water (%)	Control Limits 8081/8082 Soil (%)	RPD Limit (608)	RPD Limit (8081/8082)
4,4'-DDD	31-141	56-143	56-139	40	30
4,4'-DDE	30-145	57-135	56-134	40	30
4,4'-DDT	25-160	51-143	50-141	40	30
Aldrin	42-122	45-134	45-136	40	30
alpha-BHC	37-134	54-138	45-137	40	30
alpha-Chlordane	55-143	60-129	54-133	40	30
Aroclor-1016 (PCB-1016)	50-114	46-129	47-134	40	30
Aroclor-1260 (PCB-1260)	8-127	45-134	53-140	40	30
beta-BHC	17-147	56-136	50-136	40	30
Chlordane (tech.)	45-119	62-140	43-149	40	30
delta-BHC	19-140	52-142	47-139	40	30
Dieldrin	36-146	60-136	56-136	40	30
Endosulfan I	45-153	62-126	53-132	40	30
Endosulfan II	0-202	52-135	53-134	40	30
Endosulfan sulfate	26-144	62-133	55-136	40	30
Endrin	30-147	60-138	57-140	40	30
Endrin aldehyde	53-186	51-132	35-137	40	30
Endrin ketone	31-169	58-134	55-136	40	30
gamma-BHC (Lindane)	32-127	59-134	49-135	40	30
gamma-Chlordane	N/A	56-136	53-135	40	30
Heptachlor	34-111	54-130	47-136	40	30
Heptachlor epoxide	37-142	61-133	52-136	40	30
Methoxychlor	29-179	54-145	52-143	40	30
Mirex	N/A	51-127	65-128	40	30
Toxaphene	N/A	33-134	33-141	40	30

Table 4: Control Limits for Surrogates

Surrogate	Control Limits 608 Water (%)	Control Limits 8081/8082 Water (%)	Control Limits 8081/8082 Soil (%)
Decachlorobiphenyl (DCB)	34-135	30-139	55-139
2,4,5,6-Tetrachloro-m-xylene (TCMX)	45-148	44-124	42-129

Table 5: Marginal Sporadic Failures

Number of Analytes	Failures Allowed
<11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

STANDARD OPERATING PROCEDURE FOR

ANALYSIS OF SEMI-VOLATILE ORGANIC COMPOUNDS (SVOC) BY GC/MS

EPA 8270D

Approved by: _____ Date: 3/14/14
Katherine Brown, Semivolatiles Supervisor

Approved by: _____ Date: 3/14/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 3/14/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 EPA Method 8270D is used to quantitatively analyze most neutral, acidic and basic (BNA) organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, ethers, aldehydes, ketones, anilines, pyridines, quinolines, aromatic nitro compounds and phenols, including nitrophenols. A list of target analytes is available in the LIMS.

1.2 Theory & Principle - Gas chromatographic analysis utilizing Mass Selective Detectors eliminates, to a great extent, the secondary column and detector for confirmation. Mass selective detectors ionize each compound to produce its own unique mass fragmentation profile. Direct injection by autosampler is the introduction method for samples to be analyzed via EPA Method 8270D. The polarity of the column phase, molecular weight and temperature determine the elution order. The end of the column is positioned so that the eluting compounds are ionized immediately. The ensuing charged ions are directed along an electronically charged quadruple to the electron multiplier, where the signal is amplified. The MSD can be used in selective ion monitoring and scan mode. In the scan mode, the detector scans for all ions in a selected range. The resulting ion fragmentation profiles are compound fingerprints. The selective ion-monitoring (SIM) mode allows for the detection of two to three selected ions per compound in each retention window resulting in lower detection limits. Isomeric compounds must also depend upon retention times as a determining factor. Semi-volatile analysis primarily involves medium to high molecular weight compounds that are readily soluble in methylene chloride. The BNA sample preparation process utilizes the very polar methylene chloride, to extract the target compounds. Helium is used as the carrier gas.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water and Solid Extracts

3.0 SUMMARY OF TEST METHOD

3.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation. BNAs are extracted using method EPA 3550C (SOP EXT-001) for solid samples, method EPA 3510C or 3520C (SOP EXT-003 and EXT-004) for aqueous samples, or method EPA 3580A (SOP EXT-038) for high-level wastes.

3.2 The Semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) equipped with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS)

connected to the gas chromatograph. Analytes eluted from the capillary column are introduced into the mass spectrometer via direct injection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using an appropriate calibration curve for the intended application.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Interferences due to contamination are monitored by analysis of a method blank performed with each batch.

6.2 Interferences with the parent (quantitation) ion may occur with co-elution and must be monitored by the analyst.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Agilent 6890N GC with a 5975 Mass Spectrometer with autosampler

8.2 Agilent 7890N GC with a 5975C Mass Spectrometer with autosampler Recommended GC Operating conditions (instrument conditions are approximate and may vary by individual instrument Adjustments are made when new columns are installed. The analyst is allowed to modify settings to optimize operating conditions):

8.2.1 Oven Program – Set initial temperature to 40°C

8.2.1.1 Rate A – Ramp at 40°C per minute to 245°C

8.2.2 Hold time – 4.0-5.0 minutes

8.2.3 Carrier Gas – Helium

8.2.4 Injection volume – 1 µL

8.2.5 Run Time – 11.9 minutes

8.3 The Agilent GC utilizes Agilent software GC Chemstation Version E.02.00.493 to control the instrument and transfer data acquisition to Target Data Integration software. Target software is utilized to calculate all data. In the event of software malfunction for the GC Chemstation, please contact Agilent technical support for troubleshooting help.

8.4 The data system must be capable of time stamping all data produced with the correct date and time. The data system employed will be Target. In the event of software malfunction when using Target, please contact GCAL's IT Director for troubleshooting help.

8.5 Restek RX1 5sil MS 30m x 0.25mm ID, 0.25µm df column

8.6 Gas-tight syringes

8.7 Volumetric Flasks

8.8 Disposable glass micropipettes

8.9 Pasteur pipets

8.10 Vials – 2 mL auto-sampler vials with Teflon lined crimp caps

8.11 Bottles – amber glass with PTFE-lined screw tops or crimp tops

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of pesticide grade or equivalent and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date for a reagent, the expiration date will be 5 years from the date manufactured or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date

opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Methylene chloride (DCM)

9.1.2 Helium gas

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared working standards as described in SOP GEN-006.

9.2.1 Tuning Standard (commercially prepared, 500 µg/mL in methylene chloride). Store in a freezer at ≤-10ºC.

9.2.2 Stock Internal Standard (IS) Mix (commercially prepared, 4000 µg/mL in methylene chloride). Store in a freezer at ≤-10ºC in glass sealed ampules.

9.2.3 Stock Calibration Standard (TCL/SS) Mix (commercially prepared, 200 µg/ml multi-analyte mix. Store in a freezer at ≤-10ºC in glass sealed ampules.

9.2.4 Stock Calibration Standard (APPIX) Mix (commercially prepared, 2000 µg/mL multi-analyte mix). Store in a freezer at ≤-10ºC in glass sealed ampules.

9.2.5 Independent stock standards – standard mixes identical to those listed in 9.2.3– 9.2.4 from a secondary source or a different lot number from the same manufacturer. These standards are used to prepare the ICV.

9.2.6 Additional Stock Standards may be purchased for project-specific requirements.

9.2.7 Working Standards – Prepare all working standards by diluting the stock standard as described below and record in the Standards Preparation Logbook. Assemble the necessary glassware, syringes and solvent. Spectroscopic grade methylene chloride is used in making all standards. Ensure that all glassware and syringes are clean and free of moisture by rinsing with at least three aliquots of methylene chloride. Volumetric glassware must never be stored in or placed in heated ovens. The use of dedicated glass will help prevent cross-contamination of standards. Prepare standards in a volumetric flask and then transfer to a 2 mL vial. Store working standards in the same manner as the stock standards. Always quantitate new standards against known standards to ensure accurate concentration levels.

9.2.7.1 Working Appendix IX standard – prepare a 200 µg/mL standard mix from the 2000 µg/mL stock standard.

9.2.8 Calibration standards – 0.1 µg/mL to 160 µg/mL calibration standards are prepared as needed before a calibration is performed using the stock 200 µg/mL TCL mix or the working 200 µg/mL APP IX mix. Calibration standards are stored in amber vials in the freezer at ≤-10ºC. Prepare 1.0 mL of each calibration standard as indicated below. The standards are diluted with Methylene Chloride (DCM).

9.2.8.1 0.2 µg/mL Standard – Dilute 1 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.2 1.0 µg/mL Standard – Dilute 5 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.3 2.0 µg/mL Standard – Dilute 10 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.4 10.0 µg/mL Standard – Dilute 50 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.5 50.0 µg/mL Standard – Dilute 250 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.6 80.0 µg/mL Standard – Dilute 400 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.7 120 µg/mL Standard – Dilute 600 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.8 160 µg/mL Standard – Dilute 800 µL of 200 µg/mL standard to a final volume of 1.0 mL with DCM.

9.2.8.9 200 µg/mL Standard – Stock TCL or Working APPIX standard

9.2.8.10 Add 10 µL of 4000 ng/µL Internal Standard Mix to each 1.0 mL calibration standard before injecting.

9.2.8.11 To make the calibration standards for SIM analysis, a 0.1 µg/mL standard is added to the above calibration levels.

9.2.8.11.1 0.1 µg/mL Standard – Dilute 10 µL of 10 µg/mL standard (9.2.8.4) to a final volume of 1.0 mL with DCM.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in Teflon-lined containers with minimal headspace and stored at the laboratory using conditions required by 40RFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation
- 11.1.1 Aqueous
  - 11.1.1.1 Cool to 0-6° C, but not frozen
- 11.1.2 Solid
  - 11.1.2.1 Cool to 0-6° C, but not frozen

11.2 Maximum holding time
- 11.2.1 Waters – 7 days from collection to extraction; 40 days from extraction to analysis at ≤-10ºC in sealed vials. Protect from light.
- 11.2.2 Solids – 14 days from collection to extraction; 40 days from extraction to analysis at ≤-10ºC in sealed vials. Protect from light.

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One LCS and/or LCS/LCSD shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to reagent water or sand and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike and matrix spike duplicate (MS/MSD) shall be analyzed per batch if sufficient sample volume is available. The MS is a known amount of standard added to the sample at the same concentration as the LCS.

12.4 Internal standards and surrogates are added to all samples, including QC samples.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Instrument Tuning Procedure

13.1.1 Before analysis begins each GC/MS must meet tuning criteria specified in the method. A solution containing 50 µg/ml each of Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, Pentachlorophenol and Benzidine is analyzed.

13.1.1.1 The DFTPP tuning criteria in Table 2 must be met before proceeding with analysis. DDT breakdown shall not exceed 20% and is calculated as:

$$\%\text{Breakdown} = \frac{\text{area DDE} + \text{area DDD}}{\text{area DDE} + \text{area DDD} + \text{area DDT}} \times 100$$

13.1.1.2 Evaluate the tailing factor for Benzidine and Pentachlorophenol. Check that response is normal and that there is not excessive tailing. If tailing is observed, calculate tailing factors using the equations in EPA 625 Figure 13. The calculated tailing factor for

Benzidine and for Pentachlorophenol shall be less than 2.0. Excess tailing indicates the need for instrument maintenance. If there is no visible tailing document this.

13.1.2 The mass spectrum of DFTPP is acquired by using the average of three scans: the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using a single scan no more than 20 scans prior to the elution of DFTPP. The tune method is hard coded to perform the above functions and is checked manually if DFTPP tune criteria fail.

13.1.3 The DFTPP tuning criteria in Table 2 must be met before proceeding with analysis. If these criteria cannot be met, the instrument should be manually tuned with FC-43. If tune criteria are still not met instrument maintenance may be performed.

13.1.4 Once the DFTPP tune criteria have been met, the instrument is considered to be tuned for a twelve-hour period starting from the injection time of the DFTPP.

13.1.5 The instrument must be retuned before the twelve-hour period has elapsed if any major adjustments are made to the mass spectrometer that may affect the tuning criteria.

13.2 Initial Calibration (ICAL) – an ICAL is performed using a blank and at least 5 standards. An initial calibration curve must be analyzed on each instrument and evaluated before any sample result can be quantitated.

13.2.1 ICAL standards are analyzed at the calibration levels listed in Tables 1A and 1B. Because nine calibration standards are used, the analyst has the option of eliminating several of the points using the criteria below. Within the following requirements the analyst may select the points that improve linearity, obtain the calibration range needed for a specific project, or maintain the default calibration range (10-200 µg/mL):

13.2.1.1 At least five contiguous points must be used (six if a quadratic curve fit is employed).

13.2.1.2 Calibration acceptance criteria must be met as described below.

13.2.1.3 The lowest calibration point must support the lowest reporting limit needed in the associated samples.

13.2.1.4 The QC spike amount must be within the calibration range.

13.2.2 Replacing points in the middle of a curve is not allowed unless the analyst can document a technical issue at the time of analysis or spiking of the standard. The new point must be analyzed in the same analytical batch. If the problem appears to be associated with a single standard, that one standard may be reanalyzed. Replacing the standard may be necessary in some cases. Prepare the calibration standards and Add 10 µL of 4000 µg/ml Internal Standard Mix to each.

13.2.3 When performing analysis in the SIM mode, use the same retention windows used in scan analysis and at least two of the ions listed in Table 1

of SW-846 8270D. The primary ion is used for quantitation and the secondary ion(s) as confirmation.

13.2.4 In addition, when performing SIM analysis the following minimum response factors should be met for the PAH compounds. The method allows for a maximum of 2 of these compounds to not meet this requirement.

Acenaphthene	0.9	Dibenz(a,h)anthracene	0.4
Acenaphthylene	0.9	Fluoranthene	0.6
Anthracene	0.7	Fluorene	0.9
Benzo(a)anthracene	0.8	Indeno(1,2,3-cd)pyrene	0.5
Benzo(a)pyrene	0.7	2-Methylnapthene	0.4
Benzo(b)fluoranthene	0.7	Naphthalene	0.7
Benzo(g,h,i)perylene	0.5	Phenathrene	0.7
Benzo(k)fluoranthene	0.7	Pyrene	0.6
Chrysene	0.7		

13.2.5 The acceptance criteria for initial calibration must be satisfied before analysis of samples begins. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.

13.2.6 Once all levels of standards have been analyzed, the validity of the initial calibration must be verified by confirming that the Minimum Response Factors (RF) listed in method 8270D Table 4 (attached) are met.

13.2.7 Additionally one of the options below must be met. Always attempt to meet calibration criteria using the average response factor. If the average response factor does not pass, options 13.2.8.2 and 13.2.8.3 are evaluated, but do not need to be evaluated in the order listed (if historical results indicate that quadratic fits are appropriate for a particular analyte, that option may be selected without evaluating linear). Option 13.2.8.4 may be used for some projects if the other options are not successful. The calibration options and requirements are as follows:

13.2.7.1 Average Response Factor Calibration. For each of the standards, calculate the response factor of each compound. Calculate the average of a minimum of five response factors and the standard deviation across the selected five response factors. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD). All equations can be found in the Calculation section. When the five (or more) response factors of the standards demonstrate less than 20% RSD for a target analytes, linearity through the origin can be assumed. If the RSD for any analyte is greater than 20%, the analyst may wish to

review the results for those analytes to ensure that the problem is not associated with just one of the initial calibration standards.

13.2.7.2 For those compounds that the RSD exceeds 20%, a linear regression equation that is not forced through the origin may be used. The coefficient of determination (r^2) must be at least 0.990 for the curve to be acceptable. As part of the evaluation, check the y-intercept (b) and any negative factors (b or M). Negative factors may result in false negatives and positives. Large shifts in the y-intercept from zero will impact the data. If the intercept is greater than half the reporting limit, this option shall not be used. Check the required reporting limit and any impacts on the data. False positives shall be indicated in the case narrative. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point shall be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point shall be within ± 30% of the standard's true concentration. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the lower limit of quantitation and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

13.2.7.3 A quadratic curve fit may be used if the coefficient of determination $r^2 \geq 0.990$. A minimum of a six-point calibration is used if this option is chosen and the curve shall not be forced through zero. As part of the evaluation, check the y-intercept (b) and any negative factors (b or M). Negative factors may result in false negatives and positives. Large shifts in the y-intercept from zero will impact the data. If the intercept is greater than half the reporting limit, this option shall not be used. Check the required reporting limit and any impacts on the data. False positives shall be indicated in the case narrative.

13.2.7.4 If the %RSD is greater than 15% for any analyte of interest, the initial calibration may still be acceptable if the following conditions are met:

13.2.7.4.1 The mean of the RSD values for <u>all</u> analytes in the calibration is ≤15%.

13.2.7.4.2 Mean RSD = <u>Sum of RSD value for each analyte</u>

Total # analytes

13.2.7.4.3 Mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.

13.2.7.4.4 The maximum RSD allowed for any compound is 50%, except for poor performers as defined in Table 4. For poor performers which exceed 50%, narrate exceedances in the final report.

Note: the use of this calibration option is not acceptable for some projects. If this option is used, include the analytes that did not pass the first three options in the report narrative.

13.2.7.5 If the calibration criteria are not met, possible corrective action includes cleaning the injection port, cutting off the first 4 inches of column or source cleaning, and adjusting the calibration range.

13.2.7.6 Check the position of the retention time by using the mid-point of an initial calibration. Check and reset as necessary after source cleaning or column maintenance.

13.3 Initial Calibration Verification (ICV) – immediately following the ICAL analyze a standard containing all target analytes prepared from a solution that is independent from the ICAL standard. It is preferred that the ICV be from a different manufacturer. If this is not available than a different lot from the same manufacturer may be used.

13.3.1 The ICV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 30% difference from the initial calibration, with the exception of compounds identified in Table 4 as a poor performer. Poor performing compounds must not exceed a 40% difference from the initial calibration.

13.3.2 For DoD projects, the ICV recovery must not exceed a 20% difference from the initial calibration.

13.3.3 If the criterion referenced above is not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved. If holding time or agreed project due dates will not be met because of ICV failure, the client must be contacted and approve of proceeding with the analysis. Note all failures in the case narrative.

13.4 Continuing Calibration Verification (CCV) – a CCV must be analyzed daily before sample analysis, unless an ICAL is performed.

13.4.1 The CCV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 20% difference from the initial calibration and the minimum RFs listed in method 8270D Table 4 (attached) must be met, with the exception of compounds identified in Table 4 as a poor performer. Poor performing compounds must not exceed a 40% difference from the initial calibration. If the calibration verification

standard fails to meet the criteria, repeat the injection of the standard. If the standard fails again, take appropriate corrective action (inspection of instrument, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.

13.4.2 The retention time for any internal standard shall not vary any more than 30 seconds from the last calibration check (12 hours). The extracted Ion Current Profile area for any internal standard must not deviate by more than a factor of two.

13.4.3 If the calibration verification standard fails to meet the criteria, repeat the injection of the standard. If the standard fails again, take appropriate corrective action (inspection of instrument, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.

14.0 PROCEDURE

14.1 Sample Analysis

14.1.1 Injection port maintenance is performed every day of use at a minimum. This includes replacement of septa and liner.

14.1.2 Samples can be analyzed after an acceptable CCV. Add 10 µL of the internal standard mixture to each extract before analysis.

14.1.3 Monitor drift by using the internal standard report in target to check the % Difference of internal area response and retention time. Internal area response must be between -50% to +100% of the internal standard response in the mid-point of the ICAL. Retention time must be within ± 0.5 minutes. If the criteria are not met reanalyze at a dilution for the affected analytes or report with a narrative. Project specific limits may apply. See SOP GEN-019.

14.2 Quantitation

14.2.1 Target analytes are qualitatively identified based on the retention time and on comparison of the analytes mass spectrum with a reference mass spectrum. A reference mass spectrum is generated using the same conditions of the method. In the reference mass spectrum three characteristic ions are identified, one of which is the primary characteristic ion. If there are not three ions, than any ion over 30% relative intensity may be used. The target is said to be present when the following criteria are met:

14.2.1.1 The intensities of the characteristic ion(s) maximize within one scan.

14.2.1.2 The retention time of the compound in the sample must be within ± 0.50 minutes of the retention time of that compound in the standard and have an RRT of ± 0.06.

14.2.1.3 The relative intensities of the characteristic ion(s) agree within 30% of the relative intensities of these ions in the reference spectrum.

14.2.1.4 Structural isomers may be identified as individual isomers if the height of the valley between them is less than 25% of the sum of the two peak heights. Otherwise they must be reported as isomeric pairs.

14.2.2 Samples are quantitated once a positive identification has been made. The quantitation will be based on the area of the primary characteristic ion's EICP and the associated internal standard. Calculations are described in the calculation section. The quantitation ion used shall be the ion listed in the reference method(s) unless there are interferences. Qualifier ions shall be used as a replacement, if possible. If the quantitation ion used is not the ion listed in the reference method, this shall be documented and the documentation stored in the lab.

14.3 If a sample response exceeds the limits of the initial calibration of the system, a dilution of the sample must be analyzed which falls within the calibration range of the instrument.

14.3.1 A portion of the sample, measured with a syringe, is diluted to one mL with DCM. Additional internal standard is added to maintain a concentration of 40µg/L. Surrogate recovery is affected and is considered diluted out of the sample with a dilution of 10X or greater. The dilution factor is recorded in Target and used to calculate analyte concentrations and to adjust reporting limits and MDLs. Multiple dilutions are required for some projects. Report multiple dilutions as required. If historical values indicate that the sample needs to be performed at multiple dilutions and all analytes must be reported at the lowest reporting limit possible, the sample extract may be prepared and performed at multiple dilutions without screening.

14.4 Manual Integrations

14.4.1 See SOP QA-010 for GCAL's Manual Integration Policy.

14.5 Tentatively Identified Compounds (TICs)

14.5.1 A TIC search, or library search, may be performed when analyzing samples or sample extracts by GC/MS. A client may request a TIC search for specific compounds that are not calibrated for, or to determine other compounds present in a sample. Generally the client will request a reporting of the 10 or 20 highest concentration "unknowns" in a sample. This is performed using a spectral match between an unknown peak and a spectral library. Unknowns are identified based on the spectral data only. In general the retention time is not considered when identifying unknowns. In addition the concentration is an estimate based on the assumption that the response factor of the unknown is 1.0. This may lead to a considerable positive or negative bias in the reported concentration.

14.5.2 The Target method is set-up using the following parameters:

14.5.2.1 Primary Library - NBS75K.1

14.5.2.2 Match Quality Threshold – 50

14.5.2.3 Remove Duplicate Matches
14.5.2.4 Background Subtraction Method - Average of Front and Rear
14.5.2.5 Minimum percent for ID - 10% of nearest Internal Standard Area
14.5.2.6 Quantitation by - Area
14.5.2.7 RF Mode - Nearest ISTD

14.5.3 The library search does not use a normalization routine. These parameters are set in the Method and are not altered unless approved by a Supervisor or Manager. Note that the selection of peaks and the calculated amount uses the peak area of the nearest internal standard, not the response of quant ion. The area is found in the area.rp report in Target. The amount of an unknown is estimated using an RF factor of 1.

14.5.4 When performing a library search select Quant Unknown Compounds in Edit Quantitation Parameters and process the data file. The software will identify unknown peaks meeting the set criteria. If the software misses a peak that in the analyst opinion appears to meet the area requirements, the analyst may add an unknown. Select Spectra – Search, then Edit – Add Unknown. Select the TIC with the best fit (highest quality match). If no match is higher than 50% select the result as "Unknown". If in the analyst's opinion the Target selected best match is incorrect, the match is changed to reflect the best match or is reported as an unknown.

14.5.5 Review the assigned match using the following guidelines:

14.5.5.1 Major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

14.5.5.2 The relative intensities of the major ions should agree with the reference spectrum within ± 30%. Major ions that show increased intensity may be due to co-eluting compounds, and ions showing decreased intensity may be due to the background subtraction. When this is encountered examine the spectrum across the peak to determine if co-elution is an issue. If necessary, alter the background subtraction program to best handle the sample matrix.

14.5.5.3 Molecular ions present in reference spectrum should be present in sample spectrum (this is usually the heaviest ion).

14.5.6 Save changes and upload results to the LIMS. Only use the Quant Unknown Compound option when a library search is requested. If a library search is added after the file is processed, copy the file using the same file number and append a "T" behind the file number. Process the file and perform the TIC search. Do not reprocess a data file that has already been uploaded to the LIMS.

14.5.7 If a specific analyte is requested to report as a library search, perform the MS library search as described. If the analyte is identified, post the result. If the analyte is not identified, post the TIC file and enter the CAS

number, target analyte, and ND in the CAS/Name column of the sample results. Leave the amount column empty.

15.0 CALCULATIONS

15.1 % Recovery (MS/MSD) = $\frac{\text{Spike Concentration-Sample Concentration}}{\text{Spike Added}} \times 100$

15.2 % Recovery (LCS/LCSD/Surrogate) = $\frac{\text{Spike Concentration}}{\text{Spike Added}} \times 100$

15.3 RPD = $\frac{|\text{Concentration 2-Concentration 1}|}{(\text{Concentration 2+Concentration 1})/2} \times 100$

15.4 Response Factor $= A_s/C_s$

As= Peak Area of analyte or surrogate

Cs= Concentration of the analyte or surrogate

15.5 % Difference = $[(RF_I - RF_C) / RF_I] \times 100$

RF_I = Average response factor from initial calibration

RF_C = Response factor from current verification check standard

15.6 % RSD = (SD/X) X 100

RSD = Relative Standard Deviation

X = mean of initial RF's for a compound

SD = Standard Deviation of average RF's for a compound

15.7 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMIT / LIMIT OF QUANTITATION (LOQ)

16.1 Water samples – L4 calibration standard listed in Table 1A

16.2 Water SIM samples – L1 calibration standard listed in Table 1B

16.3 Solid samples – water LOQ * prep factor

Where prep factor = final volume (mL)/initial weight (g)

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in method 8270D.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 20 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 10% for CCCs. This shall be evaluated quarterly. Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the on-column amount measured in any sample (whichever is greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code “RP” for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately.

19.2 The LCS control limits specified in Tables 3A and 3B are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria, the source of inaccuracy should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code “RP” for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.3 The MS is evaluated against the LCS control limits and is used to determine sample matrix effect. If the MS recovery is outside of control limits and the LCS is within control limits, matrix interference is indicated. Report the results with a case narrative.

19.4 The LCS/LCSD and MS/MSD RPD criteria are specified in Tables 3A and 3B. If the RPD is outside the control limits, investigate the source of the precision error. A source of precision error in the MS/MSD may be the heterogenous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.5 The surrogate criteria are specified in Tables 5A and 5B. If a surrogate recovery is outside QC limits in a MB, LCS, or LCSD, the entire batch must be re-extracted and/or reanalyzed. Enter condition code "RP" for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If more than one surrogate per fraction (acid or base/neutral) is outside QC limits, or if any surrogate is less than 10% in a sample and no obvious matrix interference is present, the sample should be re-extracted and/or reanalyzed. If the surrogate recovery is confirmed report the original data prep and analysis. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If obvious chromatographic interference with surrogate is present, reanalysis is not necessary. Report the data with a case narrative. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.6 Internal Standards (IS)

19.6.1 The criteria for internal standards for QC samples (ICV, CCV, MB, LCS, LCSD) is -50% to +100% of the daily CCV. QC samples may not be reported if IS criteria is not met.

19.6.2 The criteria for internal standards for investigative samples is -50% to +100% of the daily CCV. For samples with IS results outside criteria, determine if the IS failure is due to instrument malfunction or sample matrix. Corrective action if IS failure is due to instrument malfunction: rerun sample undiluted (or same dilution as original analysis). Corrective action if IS failure is due to sample matrix: run compounds that fail criteria at a 5x dilution, or use the methanol extract for soil samples.

19.6.3 For DoD projects, the criteria for internal standards for QC samples (ICV, CCV, MB, LCS, LCSD) and investigative samples is -50% to +100% of the ICAL midpoint.

19.6.4 The retention time of the IS in the sample must not shift greater than + 0.5 min of the retention time of the corresponding IS in the CCV.

19.7 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.8 Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative. Enter condition code "DL" in the LIMS for the affected surrogates.

19.9 Spikes may be diluted out in the analysis process if a 10X or higher dilution is performed. Document this in the case narrative.

19.10 Project specific criteria may apply. See SOP GEN-019.

19.11 For data review procedures and checklists, see SOP QA-002.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 8000B

22.2 EPA 8270D

23.0 METHOD MODIFICATIONS

23.1 None

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Table 1A: Scan Calibration Concentrations

24.3 Table 1B: SIM Calibration Concentrations

24.4 Table 2: DFTPP Tuning Acceptance Criteria

24.5 Table 3A: Scan Control Limits for LCS/LCSD/MS/MSD and IS Associations

24.6 Table 3B: SIM Control Limits for LCS/LCSD/MS/MSD and IS Associations

24.7 Table 4: Poor Performing Compounds

24.8 Table 5A: Scan Control Limits for Surrogates

24.9 Table 5B: SIM Control Limits for Surrogates

24.10 Table 6: Criteria for Marginal Sporadic Failures

24.11 Attachment 1 – Method 8270D Table 4, Minimum Response Factors for ICAL

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
01/25/13	08	Previous version	
03/14/14	09	Updated format and added sections required for TNI-compliant SOPs. Added Tables, including control limits and internal standard associations. Updated SOP to reflect the same spike and surrogate concentrations for scan and SIM analysis. Updated control limits.	Shelley Bourgeois Katherine Brown

Table 1A: Scan Calibration Concentrations, (µg/mL)

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	L9
Analyte concentration unless specified	0.2	1.0	2	10	50	80	120	160	200

Table 1A: SIM Calibration Concentrations, (µg/mL)

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	L9
Analyte concentration (all PAHs)	0.1	1.0	2	10	50	80	120	160	200

Table 2: DFTPP Acceptance Criteria

M/Z	ION ABUNDANCE CRITERIA
51	30 TO 60% of mass 198
68	less than 2% of mass 69
69	mass 69 relative abundance
70	less than 2% of mass 69
127	40 to 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	greater than 1% of mass 198
441	present but less than mass 443
442	greater than 40% of mass 198
443	17 to 23% of mass 442

Table 3A: Control Limits for LCS/LCSD/MS/MSD and Internal Standard Associations

Analyte	Associated Internal Standard	Control Limits, Water (%)	Control Limits, Soil (%)	RPD Limit
1,2,4,5-Tetrachlorobenzene	3	35-121	37-119	30
1,2,4-Trichlorobenzene	2	29-116	34-118	30
1,2-Dichlorobenzene	1	32-111	33-117	30
1,2-Diphenylhydrazine/Azobenzene	4	49-122	41-125	30
1,3,5-Trinitrobenzene	4	41-137	57-121	30
1,3-Dichlorobenzene	1	28-110	30-115	30
1,3-Dinitrobenzene	3	49-128	43-127	30
1,4-Dichlorobenzene	1	29-112	31-115	30

1,4-Dioxane	1	40-120	10-130	30
1,4-Naphthoquinone	3	20-120	55-108	30
1,4-Phenylenediamine	2	10-120	10-120	30
1-Methylnaphthalene	2	41-119	40-119	30
1-Naphthylamine	3	24-124	20-120	30
2,3,4,6-Tetrachlorophenol	3	50-128	44-125	30
2,4,5-Trichlorophenol	3	53-123	41-124	30
2,4,6-Trichlorophenol	3	50-125	39-126	30
2,4-Dichlorophenol	2	47-121	40-122	30
2,4-Dimethylphenol	2	31-124	30-127	30
2,4-Dinitrophenol	3	23-143	14-120	30
2,4-Dinitrotoluene	3	57-128	48-126	30
2,6-Dichlorophenol	2	50-118	41-117	30
2,6-Dinitrotoluene	3	57-124	46-124	30
2-Acetylaminofluorene	5	60-138	54-134	30
2-Chloronaphthalene	3	40-116	41-114	30
2-Chlorophenol	1	38-117	34-121	30
2-Methylnaphthalene	2	40-121	38-122	30
2-Naphthylamine	3	20-120	20-120	30
2-Nitroaniline	3	55-127	44-127	30
2-Nitrophenol	2	47-123	36-123	30
2-Picoline	1	34-109	27-103	30
3,3'-Dichlorobenzidine	5	27-129	22-121	30
3-Methylcholanthrene	6	56-133	56-134	30
3-Nitroaniline	3	41-128	33-119	30
4,6-Dinitro-2-methylphenol	4	44-137	29-132	30
4-Aminobiphenyl	4	20-120	20-120	30
4-Bromophenyl phenyl ether	4	55-124	46-124	30
4-Chloro-3-methylphenol	2	52-119	45-122	30
4-Chloroaniline	2	33-117	17-106	30
4-Chlorophenyl phenyl ether	3	53-121	45-121	30
4-Nitroaniline	3	38-120	32-120	30
4-Nitrophenol	3	10-120	30-132	30
4-Nitroquinoline 1-oxide	4	20-120	20-120	30
5-Nitro-o-toluidine	3	38-126	23-117	30
7,12-Dimethylbenz(a)anthracene	6	61-133	50-142	30
Acenaphthene	3	47-122	40-123	30

Acenaphthylene	3	41-130	32-132	30
Acetophenone	2	46-118	33-115	30
Aniline	1	19-124	21-131	30
Anthracene	4	57-123	47-123	30
Aramite	5	34-132	20-120	30
Atrazine	4	44-142	47-127	30
Benzaldehyde	1	40-118	25-127	30
Benzidine	5	10-120	10-120	30
Benzo(a)anthracene	5	58-125	49-126	30
Benzo(a)pyrene	6	54-128	45-129	30
Benzo(b)fluoranthene	6	53-131	45-132	30
Benzo(g,h,i)perylene	6	50-134	43-134	30
Benzo(k)fluoranthene	6	57-129	47-132	30
Benzoic acid	2	10-120	14-124	30
Benzyl alcohol	1	31-112	29-122	30
Biphenyl	2	49-115	40-117	30
Bis(2-Chloroethoxy)methane	2	48-120	36-121	30
Bis(2-Chloroethyl)ether	1	43-118	31-120	30
Bis(2-Chloroisopropyl)ether	1	37-130	33-131	30
Bis(2-Ethylhexyl)phthalate	5	55-135	51-133	30
Butyl benzyl phthalate	5	53-134	48-132	30
Caprolactam	2	10-120	46-117	30
Carbazole	4	60-122	50-123	30
Chlorobenzilate	5	58-150	49-150	30
Chrysene	5	59-123	50-124	30
Diallate	4	67-124	56-132	30
Dibenz(a,h)anthracene	6	51-134	45-134	30
Dibenzofuran	3	53-118	44-120	30
Diethyl phthalate	3	56-125	50-124	30
Dimethoate	4	20-120	28-108	30
Dimethyl phthalate	3	45-127	48-124	30
Di-n-butyl phthalate	4	59-127	51-128	30
Di-n-octyl phthalate	6	51-140	45-140	30
Dinoseb	4	61-126	16-119	30
Diphenylamine	4	55-111	48-111	30
Disulfoton	4	55-130	20-120	30
Ethyl methanesulfonate	1	62-118	34-136	30

Ethyl Parathion (Parathion)	4	66-140	20-120	30
Famphur	5	10-120	10-120	30
Fluoranthene	4	57-128	50-127	30
Fluorene	3	52-124	43-125	30
Hexachlorobenzene	4	53-125	45-122	30
Hexachlorobutadiene	2	22-124	32-123	30
Hexachlorocyclopentadiene	3	16-120	23-121	30
Hexachloroethane	1	21-115	28-117	30
Hexachloropropene	2	38-120	32-132	30
Indeno(1,2,3-cd)pyrene	6	52-134	45-133	30
Isodrin	4	68-128	56-132	30
Isophorone	2	42-124	30-122	30
Isosafrole	2	56-126	43-136	30
Kepone	4	10-120	10-120	30
m,p-Cresol	1	29-110	34-119	30
Methapyrilene	4	20-120	20-120	30
Methyl methanesulfonate	1	33-107	38-117	30
Methyl parathion	4	45-159	20-120	30
Naphthalene	2	40-121	35-123	30
n-Decane	1	29-105	N/A	30
Nitrobenzene	2	45-121	34-122	30
N-Nitrosodiethylamine	1	43-121	41-124	30
N-Nitrosodimethylamine	1	12-120	23-120	30
N-Nitrosodi-n-butylamine	2	60-121	59-124	30
N-Nitrosodi-n-propylamine	1	49-119	36-120	30
N-Nitrosodiphenylamine	4	51-123	38-127	30
N-Nitrosomethylethylamine	1	41-117	34-123	30
N-Nitrosomorpholine	1	55-117	50-133	30
N-Nitrosopiperidine	2	56-121	60-119	30
N-Nitrosopyrrolidine	1	48-113	45-126	30
n-Octadecane	1	50-128	44-131	30
o and/or p-Toluidine	1	30-110	20-120	30
O,O,O-Triethyl phosphorothioate	2	66-119	59-124	30
o-Cresol	1	30-117	32-122	30
p-Dimethylaminoazobenzene	5	62-132	64-134	30
Pentachlorobenzene	3	59-123	54-125	30
Pentachloroethane	1	30-92	39-102	30

Pentachlorophenol	4	35-138	25-133	30
Pentchloronitrobenzene	4	54-135	38-134	30
Phenacetin	4	71-124	57-133	30
Phenanthrene	4	59-120	50-121	30
Phenol	1	16-120	34-121	30
Phorate	4	38-139	20-120	30
Pronamide	4	65-129	56-130	30
Pyrene	5	57-126	47-127	30
Pyridine	1	10-120	11-120	30
Safrole	3	61-119	47-129	30
Sulfotepp	3	61-133	52-137	30
Thionazine	3	72-132	62-127	30
Total Cresols	1	24-120	46-120	30

Internal Standards:	1	1,4-Dichlorobenzene-d_4
	2	Naphthalene-d_8
	3	Acenaphthene-d_{10}
	4	Phenanthrene-d_{10}
	5	Chrysene-d_{12}
	6	Perylene-d_{12}

Table 3B: Control Limits for LCS/LCSD/MS/MSD and Internal Standard Associations (SIM Analysis)

Analyte	Associated Internal Standard	Control Limits, Water (%)	Control Limits, Soil (%)	RPD Limit
1-Methylnaphthalene	2	41-115	43-111	30
2-Methylnaphthalene	2	39-114	39-114	30
Acenaphthene	3	48-114	44-111	30
Acenaphthylene	3	35-121	39-116	30
Anthracene	4	53-119	50-114	30
Benzo(a)anthracene	5	59-120	54-122	30
Benzo(a)pyrene	6	53-120	50-125	30
Benzo(b)fluoranthene	6	53-126	53-128	30
Benzo(g,h,i)perylene	6	44-128	49-127	30
Benzo(k)fluoranthene	6	54-125	56-123	30

Chrysene	5	57-120	57-118	30
Dibenz(a,h)anthracene	6	44-131	50-129	30
Fluoranthene	4	58-120	55-119	30
Fluorene	3	50-118	47-114	30
Indeno(1,2,3-cd)pyrene	6	48-130	49-130	30
Naphthalene	2	43-114	38-111	30
Phenanthrene	4	53-115	49-113	30
Pyrene	5	53-121	55-117	30

Internal Standards:	1	1,4-Dichlorobenzene-d_4
	2	Naphthalene-d_8
	3	Acenaphthene-d_{10}
	4	Phenanthrene-d_{10}
	5	Chrysene-d_{12}
	6	Perylene-d_{12}

Table 4: Poor Performing Compounds

Benzidine	1,4-Naphthoquinone	4-Nitroquinoline-1-oxide
Benzoic Acid	m-Dinitrobenzene	Parathion
2-Picoline	Pentachlorobenzene	Metapyriline
n-Nitrosomethylethylamine	2-Naphthylamine	Isodrin
Methyl methanesulfonate	2,3,4,6-Tetrachlorophenol	Aramite
n-Nitrosodiethylamine	1-Naphthylamine	p-(Dimethylamino) azobenznen
Ethyl methanesulfonate	Thionazin	Chlorobenzilate
Pentachloroethane	5-Nitro-o-toluidine	Famphur
o-Toluidine	Tetraethyl dithiopyrophosphate	3,3'-Dimethylbenzidine
n-Nitrosomorpholine	Diallate	2-Acetylaminofluorene
n-Nitrosopiperidine	Phorate	7,12-Dimethylbenz(a)anthracene
o,o,o-Triethylphosphoroate	sym-Trinitrobenzene	Hexachlorophene
α,α-Dimethylphenethylamine	Phenacetin	3-Methylcholanthrene
Hexachloropropene	Dimethoate	Acrylamide
2,6-Dichlorophenol	Pentachloronitrobenzene	n-Nitrosopyrrolidine
n-Nitrosodi-n-butylamine	4-Aminobiphenyl	Phthalic Acid/Anhydride
p-Phenylenediamine	Pronamide	1,4-Dinitrobenzene
Isosafrole	Dinoseb	Kepone

1,2,4,5-Tetrachlorobenzene	Disulfoton	4,4-Methylenebis(2-chloroaniline)
Safrole	Methyl Parathion	tris-2,3-Dibromopropylphosphate
Maleic Anhydride	Benzaldehyde	

Table 5A: Control Limits for Surrogates

Surrogate	Control Limits, Water (%)	Control Limits, Soil (%)
2-Fluorophenol	19-119	35-115
Phenol-d5	10-120	33-122
2,4,6-Tribromophenol	40-140	39-132
Nitrobenzene-d5	44-120	37-122
2-Fluorobiphenyl	44-119	44-115
Terphenyl-d14	50-134	54-127

Table 5B: Control Limits for Surrogates (SIM Analysis)

Surrogate	Control Limits, Water (%)	Control Limits, Soil (%)
Nitrobenzene-d5	55-111	44-125
2-Fluorobiphenyl	53-106	46-115
Terphenyl-d14	58-132	58-133

Table 6: Marginal Sporadic Failures

Number of Analytes	Failures Allowed
<11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

TABLE 4

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

TABLE 4
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800

TABLE 4
(continued)

Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

STANDARD OPERATING PROCEDURE FOR

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS (VOC) BY GC/MS

SW-846 8260B

Approved by: _____ Date: 10/03/13
Chris Harris, Volatiles Supervisor

Approved by: _____ Date: 10/03/13
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 10/03/13
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This SOP provides procedures for the gas chromatography/mass spectrometry (GC/MS) analysis of volatile organic compounds. An aliquot of sample is introduced into the GC/MS using purge and trap methods 5030B for aqueous samples or 5035 for solid and waste samples and analyzed by method 8260B.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water, solid, and waste

3.0 SUMMARY OF TEST METHOD

3.1 A 5 mL aliquot of sample is purged with nitrogen at ambient temperature. The purgeable compounds are transferred to the vapor phase and are carried to the trap. Once the purging has been completed, the trap is heated and back flushed with nitrogen to desorb the purgeable compounds onto the column. The compounds are then separated by their molecular weight, and then detected by a mass spectrometer.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware, and other sample artifacts may interfere with sample analysis. Interferences are monitored by the analysis of a method blank performed with each batch.

6.2 Samples can be contaminated by diffusion of vapors through the septum seal during shipment and storage. A field blank can be prepared during sample collection and can act as a check for this type of contamination. Holding blanks are analyzed every two weeks to provide an additional check for this type of contamination. These holding blanks are stored in coolers C04, C22, C30, and C51.

6.3 Contamination can also be seen when a low level sample is analyzed immediately after a high level sample. Freedom from contamination must be established before sample analysis is considered valid. After analysis of a sample containing high concentrations of volatile organic compounds, a blank should be analyzed to check for contamination. Alternatively, if the sample following the high

concentration sample does not contain the volatile organic compounds present in the high level sample above quantitation limits, freedom from contamination has been established. If the blank or preceding sample shows contamination, analyze blanks and/or clean the purge vessel and bake trap until an acceptable blank is achieved. Additional instrument maintenance may be required. Reanalyze samples with potential carryover contamination.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Agilent 6890 and 7890 GC with a 5973 or 5975 Mass Spectrometer coupled with one of the purge and trap/autosamplers listed below

8.2 Agilent 5890 GC with a 5971A or 5972 Mass Spectrometer coupled with one of the purge and trap/autosamplers listed below

8.3 Teledyne Tekmar Velocity XPT purge and trap

8.4 Teledyne Tekmar Solatek 72 autosampler

8.5 ATOMX purge and trap and autosampler

8.6 Recommended GC/Purge and Trap Operating conditions (instrument conditions are approximate and may vary by individual instrument):

- 8.6.1 Oven Program – Set initial temperature to 40°C
  - 8.6.1.1 Rate A – Ramp at 15-20°C per minute to 100°C
  - 8.6.1.2 Rate B – Ramp at 25-30°C per minute to 225°C
- 8.6.2 Hold time – 1.5-4.0 minutes
- 8.6.3 Carrier Gas – Helium
- 8.6.4 Purge Gas – Nitrogen
- 8.6.5 Purge volume – 5 mL

8.6.6 Purge – 11 minutes at 30°C
8.6.7 Desorb Preheat – 245°C
8.6.8 Desorb – 0.5-1.5 minutes at 250°C
8.6.9 Bake – 4 minutes at 260°C

8.7 The Agilent GC utilizes Agilent software GC Chemstation Version E.02.00.493 to control the instrument and transfer data acquisition to Target Data Integration software. Target software is utilized to calculate all data. In the event of software malfunction for the GC Chemstation, please contact Agilent technical support for troubleshooting help.

8.8 The data system must be capable of time stamping all data produced with the correct date and time. The data system employed will be Target. In the event of software malfunction when using Target, please contact GCAL's IT Director for troubleshooting help.

8.9 Restek RTX-VMS 30m x 0.25mm ID, 1.4 um df Column

8.10 Gas-tight syringes

8.11 Analytical balance – capable of weighing to 0.1g and calibrated daily when in use

8.12 Vials – 2 mL mininert vials, 2 mL auto-sampler vials with Teflon lined crimp caps, 5 mL vials with Teflon lined cap, and 44 mL VOA vials

8.13 Volumetric Flasks

8.14 Pasteur pipets

8.15 Magnetic stir bars

8.16 Scoopula spatulas

8.17 pH paper

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all organic reagent and solvent grades must be of Purge & Trap grade or equivalent and meet method requirements. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Reagent water
9.1.2 Methanol, Purge & Trap grade or equivalent
9.1.3 Sand (sea sand or equivalent)
9.1.4 Sodium bisulfate

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's

instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock Internal Standard/Surrogate Standard (IS/SS) Mix (commercially prepared, 5000 mg/L) – store in a freezer at ≤ -10 °C in glass sealed ampules until use. Expires according to the manufacturer's expiration date or 1 year after receipt whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.2 IS/SS Working Solution Mix (50 mg/L) – Rinse a clean 250 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 2.5 mL of methanol and discard. Using a syringe, add 2.5 mL of stock 5000 mg/L IS/SS standard to the volumetric flask. Insert a glass stopper and mix by inverting three times. Record in the Standards Preparation Logbook. Transfer into 44 mL VOA vial(s) with Teflon lined cap, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date whichever occurs first. Store in a freezer at ≤ -10 °C until use.

9.2.3 Stock Standards (commercially prepared) – store in a freezer at ≤ -10 °C in glass sealed ampules until use. Expires according to the manufacturer's expiration date or 1 year after receipt whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.3.1 8260, multi-analyte mix, 2000 mg/L (Ultra Part # DWM-588 or equivalent)

9.2.3.2 8260 Ketones, multi-analyte mix, 2000 mg/L (Ultra Part # CUS 12327 or equivalent)

9.2.3.3 8260 Custom, multi-analyte mix, 2000 mg/L (Ultra Part # CUS 12326 or equivalent)

9.2.3.4 CVE, 2-Chloroethylvinyl ether, 1000 mg/L (NSI Part # 17-04-15 or equivalent)

9.2.3.5 Appendix IX Mix 1, multi-analyte mix, 2500 mg/L (Ultra Part # CUS 12032 or equivalent)

9.2.3.6 Appendix IX Mix 2, multi-analyte mix, various concentrations (Ultra Part # CUS 11407 or equivalent)

9.2.3.7 Appendix IX Mix 3, Tetrahydrofuran, 1000 mg/L (Absolute Part # 70380 or equivalent)

9.2.3.8 Appendix IX Mix 4, 1,3-Butadiene, 2500 mg/L (Absolute Part # 93016 or equivalent)

9.2.3.9 Oxygenate Mix 1, multi-analyte mix, 2000 mg/L (Ultra Part # CUS 11651 or equivalent)

9.2.3.10 Oxygenate Mix 2, BTEX, 2500 mg/L (Ultra Part # CUS 11676 or equivalent)

9.2.3.11 Oxygenate Mix 3, Ethanol, 250000 mg/L (Ultra Part # CUS 11389 or equivalent)

9.2.3.12 Oxygenate Mix 4, 3,3-Dimethylbutanol, 25000 mg/L (Ultra Part # CUS 11390 or equivalent)

9.2.3.13 DuPont standard, multi-analyte mix, 2000 mg/L (NSI Part # Q-3160 or equivalent)

9.2.4 Stock Standards (commercially prepared) – store refrigerated at 0-6 °C in glass sealed ampules until use. Expires according to the manufacturer's expiration date or 1 year after receipt whichever is sooner or if comparisons with quality control check samples indicate a problem.

9.2.4.1 AAV mix, Acrolein and Acrylonitrile, 2000 mg/L; Vinyl acetate, 400 mg/L (NSI Part # Q2143 or equivalent)

9.2.5 Additional Stock Standards may be purchased for project-specific requirements.

9.2.6 Independent Stock Standards (commercially prepared) – standard mixes at the same concentrations above from a different manufacturer. If this is not available then a different lot from the same manufacturer may be used.

9.2.7 Working Standards – Prepare all working standards as described below and record in the Standards Preparation Logbook. Transfer into a 2 mL mininert vial with minimum headspace, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 14 days from the date of preparation, 7 days after opening, or manufacturer's expiration date, whichever occurs first. Standards should be stored in a freezer at ≤ -10 °C.

9.2.7.1 8260 Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 3.75 mL of methanol and discard. Using a syringe, add 1.25 mL each of stock 8260, 8260 ketone, and 8260 custom standards to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8 Working Standards – Prepare all working standards as described below and record in the Standards Preparation Logbook. Transfer into a 5 mL vial with minimum headspace, label with the standard name, standard ID, date prepared and expiration date. The solution will expire 6 months from the date of preparation or manufacturer's expiration date, whichever occurs first. Standards prepared in methanol should be stored in a freezer at ≤ -10 °C. Standards prepared in reagent water should be stored refrigerated at 0-6 °C until use.

9.2.8.1 CVE Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill

the flask to volume with methanol. Remove 2.5 mL of methanol and discard. Using a syringe, add 2.5 mL of stock CVE standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.2 AAV Working Standard (250/50 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with reagent water. Fill the flask to volume with water. Remove 1.25 mL of water and discard. Using a syringe, add 1.25 mL of stock AAV standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.3 Appendix IX Mix 1 Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 2.0 mL of methanol and discard. Using a syringe, add 1.0 mL of APP IX mix 1 stock standard and 1.0 mL of APP IX mix 4 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.4 Appendix IX Mix 2 Working Standard (various concentrations) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 2.5 mL of methanol and discard. Using a syringe, add 2.5 mL of APP IX mix 2 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.5 Appendix IX Mix 3 Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 2.5 mL of methanol and discard. Using a syringe, add 2.5 mL of APP IX mix 3 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.6 Oxygenates Mix 1 Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 1.25 mL of methanol and discard. Using a syringe, add 1.25 mL of Oxygenate mix 1 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.7 Oxygenates Mix 2 Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 1.0 mL of methanol and discard. Using a syringe, add 1.0 mL of Oxygenate mix 2 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.8 Oxygenates Mix 3 Working Standard (50000 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of

methanol. Fill the flask to volume with methanol. Remove 2.0 mL of methanol and discard. Using a syringe, add 2.0 mL of Oxygenate mix 3 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.9 Oxygenates Mix 4 Working Standard (5000 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 2.0 mL of methanol and discard. Using a syringe, add 2.0 mL of Oxygenate mix 4 stock standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.8.10 DuPont Working Standard (250 mg/L) – Rinse a clean 10 mL volumetric flask and syringe with three aliquots of methanol. Fill the flask to volume with methanol. Remove 1.25 mL of methanol and discard. Using a syringe, add 1.25 mL of stock DuPont standard to the volumetric flask. Insert a glass stopper and mix by inverting three times.

9.2.9 Calibration Standards – The calibration standards are prepared by diluting the working standard into at least 5 working levels. Table 1 indicates the calibration concentrations. The standards are diluted with reagent water.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in Teflon-lined containers with minimal headspace and stored at the laboratory using conditions required by 40RFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation

11.1.1 Aqueous

11.1.1.1 Cool to 0-6° C, but not frozen

11.1.1.2 HCl to pH ≤ 2

11.1.1.3 NaS_2O_3 (sodium thiosulfate) – used if requested by the client or if the samples are known to contain residual chlorine

11.1.1.4 Unpreserved – if requested by the client

11.1.1.5 Note: samples analyzed for 2-Chloroethylvinyl ether should be collected in unpreserved vials. If preserved containers are requested by the client, report with a case narrative.

11.1.1.6 Note: samples analyzed for Acrolein and Acrylonitrile should be collected in unpreserved vials and preserved in the field to pH 4-5 by drop-wise addition of acid. If preserved containers are requested by the client, report with a case narrative.

11.1.2 Solid

11.1.2.1 Cool to 0-6° C, but not frozen, except TerraCore samplers with DI water

11.1.2.2 TerraCore Samplers with DI water are frozen within 48 hours of sampling

11.1.2.3 EnCore samplers – preserved or frozen within 48 hours of sampling

11.1.2.4 TerraCore samplers – preserved in the field with sodium bisulfate or water (low-level) and methanol (medium)

11.1.2.5 Unpreserved, bulk jar – if requested by the client

11.2 Maximum holding time

11.2.1 Waters – 7 days from collection to analysis if unpreserved; 14 days from collection to analysis if preserved to pH <2.

11.2.2 Solids – 14 days from collection to analysis.

11.2.3 EnCore samplers must be preserved or frozen within 48 hours of collection.

11.2.4 TerraCore samplers with vials preserved with water must be analyzed or frozen within 48 hours of collection.

11.2.5 Note: Water samples must be checked for pH preservation after analysis unless a vial was provided for a pH check at sample receiving. Record the pH on the batch sheet. If the sample has a pH greater than 2 and the sample was analyzed after 7 days, report the results with a case narrative.

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One LCS and/or LCS/LCSD shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to reagent water or sand and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike and matrix spike duplicate (MS/MSD) shall be analyzed per batch if sufficient sample volume is available. The MS is a known amount of standard added to the sample at the same concentration as the LCS.

12.4 Internal standards and surrogates are added to all samples, including QC samples. The IS/SS solution is added automatically by the Solatek autosampler. All IS and SS compounds are spiked at 50 µg/L.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Instrument Tuning Procedure

13.1.1 The instrument should be tuned using FC-43 (perfluorotributylamine) such that all tuning criteria for 4-Bromoflurorobenzene (BFB) can be met (Table 2). Before analysis begins, each instrument must be tuned and checked by direct injecting 1 µL of a solution containing 50 mg/L BFB or by purging an amount equivalent to 50 ng of BFB standard.

13.1.2 The mass spectrum of BFB is acquired by using the average of three scans: the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using a single scan no more than 20 scans prior to the elution of BFB. The tune method is hard coded to perform the above functions and is checked manually if BFB tune criteria fail.

13.1.3 The BFB tuning criteria in Table 2 must be met before proceeding with analysis. If these criteria cannot be met, the instrument should be manually tuned with FC-43. If tune criteria are still not met instrument maintenance may be performed.

13.1.4 Once the BFB tune criteria have been met, the instrument is considered to be tuned for a twelve-hour period starting from the injection time of the BFB.

13.1.5 The instrument must be retuned before the twelve-hour period has elapsed if any major adjustments are made to the mass spectrometer that may affect the tuning criteria.

13.2 Initial Calibration (ICAL) – an ICAL is performed using a blank and at least 5 standards. An initial calibration curve must be analyzed on each instrument and evaluated before any sample result can be quantitated.

13.2.1 ICAL standards are analyzed at the calibration levels listed in Tables 1A and 1B. These are the routine concentrations analyzed. In all cases the standards shall be run from lowest concentration to highest, or a blank shall be run following a high concentration standard (standards greater than 50 ppb) to establish a contaminant free instrument. Once all levels of standards have been analyzed, the validity of the initial calibration must be verified by comparing the CCCs and SPCCs to the following criteria.

13.2.1.1 Calibration check compounds (CCCs) listed below must be ≤ 30 % RSD.

13.2.1.1.1 1,1-Dichloroethene
13.2.1.1.2 Chloroform
13.2.1.1.3 1,2-Dichloropropane
13.2.1.1.4 Toluene
13.2.1.1.5 Ethylbenzene
13.2.1.1.6 Vinyl Chloride

13.2.1.2 System Performance Check Compounds (SPCCs) must meet the minimum response factor (RF) requirement indicated below. All SPCCs must be evaluated at each level of the calibration curve to insure that compound instability and degradation are checked as required by the method.

13.2.1.2.1 Chloromethane – minimum RF 0.1
13.2.1.2.2 1,1-Dichloroethane – minimum RF 0.1
13.2.1.2.3 Bromoform – minimum RF 0.1
13.2.1.2.4 1,1,2,2-Tetrachloroethane – minimum RF 0.3
13.2.1.2.5 Chlorobenzene – minimum RF 0.3

13.2.2 Average Response Factor Calibration

13.2.2.1 A minimum of five standards is required for this method. Calculate the response factor (RF) of each compound using Equation 15.4.

13.2.2.2 Calculate the average of the response factors and the standard deviation across the response factors. The lowest standard used must be at or below the reporting limit for each analyte. Use the average RF and the standard deviation to calculate the percent relative standard deviation (%RSD). When the response factors of the standards demonstrate less than 15% RSD for any target analyte, linearity through the origin can be assumed. If the RSD for any target analyte is greater than 15%, the analyst may wish to review the results for that analyte to ensure that the problem is not associated with just one of the initial calibration standards. Points may be dropped from the high and/or low end of the calibration curve if the minimum number of points and the project required detection limits are maintained. Points may not be removed from the middle of a calibration unless there is a documented reason. That point may be re-analyzed and replaced in the same analytical batch.

13.2.3 Linear Regression Calibration

13.2.3.1 For those compounds that the RSD exceeds 15%, a linear regression equation that is not forced through the origin may be used. The correlation coefficient must be ≥ 0.990 for the curve to be acceptable. The analyst should check the y-intercept (b). If the intercept is greater than half the reporting limit, this option should not be used.

13.2.4 Quadratic Curve Fit

13.2.4.1 A quadratic curve fit may be used if the coefficient of determination r2 ≥ 0.990. A minimum of a six-point calibration is used if this option is chosen and the curve shall not be forced through zero. The analyst should check the y-intercept (b). If the intercept is greater than half the reporting limit, this option

should not be used. For South Carolina projects, quadratic curve fits are not allowed.

13.3 Independent Calibration Verification (ICV) – immediately following the ICAL analyze a standard containing all target analytes prepared from a solution that is independent from the ICAL standard. It is preferred that the ICV be from a different manufacturer. If this is not available than a different lot from the same manufacturer may be used.

13.3.1 The ICV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 30% difference from the initial calibration, with the exception of compounds identified in Table 3 as a poor performer. Poor performing compounds must not exceed a 40% difference from the initial calibration.

13.3.2 For DoD projects, the ICV recovery must not exceed a 20% difference from the initial calibration.

13.3.3 If the criterion referenced above is not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved.

13.4 Continuing Calibration Verification (CCV) – a CCV must be analyzed before sample analysis.

13.4.1 The CCV is prepared at the same concentration as the ICAL mid-level standard, and must not exceed a 30% difference from the initial calibration, with the exception of compounds identified in Table 3 as a poor performer. Poor performing compounds must not exceed a 40% difference from the initial calibration. CCC compounds (identified in 13.3.1.1) must not exceed a 20% difference from the initial calibration.

13.4.2 If the calibration verification standard fails to meet the criteria, repeat the injection of the standard. If the standard fails again, take appropriate corrective action (inspection of instrument, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.

14.0 PROCEDURE

14.1 Preparation of EnCores

14.1.1 Preservation – A 200 g/L sodium bisulfate solution is prepared by weighing 200 g of sodium bisulfate and bringing this to volume with reagent water, in a 1L volumetric flask. 5 mL of this solution is added to a VOA vial and a magnetic stirring bar. Record the lot # of the sodium bisulfate solution in the sample prep logbook. Tare the balance. Dispense the contents of the EnCore sampler into the vial and record the weight of the sample.

14.2 Chemstation

14.2.1 Due to shift overlaps which creates the probability that more than one analyst will be accessing a given instrument during a 12-hour tune sample

batch, GCAL utilizes a master username and password to access Chemstation software.

14.2.2 To log in to Chemstation, enter "gcmsv" for the user name and enter the password (obtained from the GCMSV supervisor) when prompted by the Chemstation software.

14.2.3 When a current password has expired, Windows prompts the user to enter current credentials including user name and password. Once this information has been entered, the user is then notified that the password has expired and must be changed. Enter a new password into the software when prompted, confirm the new password, and notify the GCMSV supervisor of the password change.

14.2.4 Chemstation use is limited to those employees who have been given password access by the GCMSV supervisor and are involved in the process of data production in the GCMSV laboratory. New employees are not granted password access until the GCMSV supervisor is satisfied that the employee has been properly trained on its use. All employees listed on current organizational chart maintained by the QA/QC department are eligible for password access if granted by the GCMSV supervisor.

14.2.5 To create an audit trail, each line in the sequencing file represents a unique sample injection. The analyst that is programming the Chemstation and loading a particular sample must include their initials in the miscellaneous information field as part of the sample sequence. This information is then transferred to Target which is used for all data acquisition. Sequence files must be saved by analytical batch and include the date in the name. On days when multiple batches are performed, letters should be added to the data to denote different analytical batches. The format for individual sample sequencing is as follows:

14.2.5.1 Type – Sample

14.2.5.2 Vial – Autosampler position

14.2.5.3 Data File – Unique sequential ascending number

14.2.5.4 Method – 8260

14.2.5.5 Sample Name – GCAL sample number

14.2.5.6 Miscellaneous Information – GCAL Batch Number*Dilution Factor*Analyst Initials

Note: only the vial, datafile, sample name, and miscellaneous information fields are edited when programming sequences. Type and method fields always remain the same for each sequence file.

14.3 Screening Samples

14.3.1 Samples may be screened by GCAL.

14.3.2 Reasons for screening may include but are not limited to the following:

14.3.2.1 Inconsistent historical data

14.3.2.2 Physical appearance of the sample

14.3.2.3 Client notification of suspected high concentration of VOCs

14.3.2.4 No historical data is available

14.3.3 Samples that are deemed necessary for screening must have adequate volume for the screening process and subsequent volume for the reportable analysis.

14.3.4 Screening data is not reportable, as the screening analysis is performed on an instrument that has not met acceptable criteria for reportable analysis. Screening data may be provided upon client request, if applicable.

14.3.5 The screening process is performed in an attempt to prevent contamination of instrumentation performing reportable analysis, thus reducing the possibility of carryover contamination from samples containing high levels of VOCs and resulting in instrument down time due to instrument maintenance.

14.3.6 Screening analyses are typically performed at a dilution factor in the range of 20 to 1000. Dilution factors may be performed outside of this range. Screening dilutions may be determined by, but are not limited to the following:

14.3.6.1 Physical appearance of the sample

14.3.6.2 Client notification of suspected high concentrations of VOCs

14.3.6.3 Historical data from similar projects

14.4 Analysis of Water Samples

14.4.1 Rinse a 50 mL volumetric flask with 3 aliquots of reagent water for each LCS, LCSD, MS, and MSD. Fill each flask with reagent water for the LCS and LCSD (the LCSD is optional, and only required if MS/MSD sample volume is limited).

14.4.2 Select a sample with sufficient volume to perform an MS and MSD. Fill each flask with sample for the MS and MSD. If sufficient sample volume is not available for an MS/MSD analysis, perform an LCS/LCSD analysis in the batch.

14.4.3 Remove the appropriate amount of reagent water from the LCS/LCSD flasks and sample from the MS/MSD flasks and then spike the standards to be analyzed at the calibration midpoint (level 7). Multiple LCS or MS samples may be prepared if the spiking solutions used contain standards which interfere or coelute. Fill a 44 mL VOA vial and immediately analyze.

14.4.4 Fill a 44 mL VOA vial with reagent water for the method blank (MB). Place the QC vials and all sample vials on the autosampler for analysis in accordance with the sample sequence. The autosampler will add the IS/SS solution.

14.5 Analysis of Soil Samples (Low-Level)

14.5.1 To prepare the spiking solution for the LCS, LCSD, MS, and MSD, rinse a 50 mL volumetric flask with 3 aliquots of reagent water and then fill with reagent water. Remove the appropriate amount of reagent water from the flask and then spike the standards to be analyzed at the calibration

midpoint (level 7). Multiple solutions may be prepared if the spiking solutions used contain standards which interfere or coelute.

14.5.2 Weigh 5.0 grams of sand into a 44 mL VOA vial for the MB, LCS, and LCSD. Add 5.0 mL of reagent water to the MB vial and 5.0 mL of spiking solution (prepared in 14.5.1) to the LCS and LCSD vials.

14.5.3 Weigh 5.0 grams of sample into a 44 mL VOA vial for each sample, MS, and MSD. Add 5.0 mL of reagent water to each sample vial and 5.0 mL of spiking solution (prepared in 14.5.1) to the MS and MSD vials.

14.5.4 Place all sample vials on the autosampler for analysis in accordance with the sample sequence. The autosampler will add the IS/SS solution.

14.6 Analysis of Soil Samples (Medium-Level)

14.6.1 Weigh 5.0 grams of sand into a 44 mL VOA vial for the MB, LCS, and LCSD. Add 5.0 mL of methanol to each vial.

14.6.2 Weigh 5.0 grams of sample into a 44 mL VOA vial for each sample, MS, and MSD. Add 5.0 mL of methanol to each sample vial.

14.6.3 For the MB and each sample, rinse a 50 mL volumetric flask with 3 aliquots of reagent water and then fill with reagent water. Remove 1.0 mL of reagent water from the each flask and add 1.0 mL of the methanol extract to the flask. Fill a 44 mL VOA vial for each sample.

14.6.4 For the LCS/LCSD and MS/MSD, rinse a 50 mL volumetric flask with 3 aliquots of reagent water and then fill with reagent water. Remove the appropriate amount of reagent water from each flask and then add 1.0 mL of the methanol extract plus the amount of spiking solution required to spike at the calibration midpoint (level 7). Multiple LCS or MS samples may be prepared if the spiking solutions used contain standards which interfere or coelute. Fill a 44 mL VOA vial for each sample.

14.6.5 Place all sample vials on the autosampler for analysis in accordance with the sample sequence. The autosampler will add the IS/SS solution.

14.7 Quantitation

14.7.1 Target analytes are qualitatively identified based on the retention time and on comparison of the analytes mass spectrum with a reference mass spectrum. A reference mass spectrum is generated using the same conditions of the method. In the reference mass spectrum three characteristic ions are identified, one of which is the primary characteristic ion. If there are not three ions, than any ion over 30% relative intensity may be used. The target is said to be present when the following criteria are met:

14.7.1.1 The intensities of the characteristic ion(s) maximize within one scan.

14.7.1.2 The RRT is within ±0.06 RRT units of the RRT of the standard.

14.7.1.3 The relative intensities of the characteristic ion(s) agree within 30% of the relative intensities of these ions in the reference spectrum.

14.7.1.4 Structural isomers may be identified as individual isomers if the height of the valley between them is less than 25% of the sum of the two peak heights.

14.7.2 Samples are quantitated once a positive identification has been made. The quantitation will be based on the area of the primary characteristic ion's EICP and the associated internal standard. Calculations are described in the calculation section. The quantitation ion used shall be the ion listed in the reference method(s) unless there are interferences. Qualifier ions shall be used as a replacement, if possible. If the quantitation ion used is not the ion listed in the reference method, this shall be documented and the documentation stored in the lab.

14.8 If a sample response exceeds the limits of the initial calibration of the system, a dilution of the sample must be analyzed which falls within the calibration range of the instrument.

14.8.1 Dilutions may be made in class A volumetric flasks. Add the approximate volume of reagent water needed to the flask. Inject the proper aliquot of the sample from a syringe into the flask. Make sure the aliquot is introduced well below the surface of the water in the flask to minimize loss of volatiles. Dilute to volume. Cap the flask and invert several times to mix. Pour the diluted sample into a 44 mL VOA vial and analyze in the same manner as any other sample.

14.8.2 Dilutions may also be made at 2x, 5x, or 10x by the instrument.

14.8.3 Dilutions may also be performed by injecting a reduced volume into the reagent water. This is the common procedure followed for methanol extracts.

14.9 Manual Integrations

14.9.1 See SOP QA-010 for GCAL's Manual Integration Policy.

14.10 Tentatively Identified Compounds (TICs)

14.10.1 A TIC search, or library search, may be performed when analyzing samples or sample extracts by GC/MS. A client may request a TIC search for specific compounds that are not calibrated for, or to determine other compounds present in a sample. Generally the client will request a reporting of the 10 or 20 highest concentration "unknowns" in a sample. This is performed using a spectral match between an unknown peak and a spectral library. Unknowns are identified based on the spectral data only. In general the retention time is not considered when identifying unknowns. In addition the concentration is an estimate based on the assumption that the response factor of the unknown is 1.0. This may lead to a considerable positive or negative bias in the reported concentration.

14.10.2 The Target method is set-up using the following parameters:

14.10.2.1 Primary Library - NBS75K.1

14.10.2.2 Match Quality Threshold - 85

14.10.2.3 Remove Duplicate Matches

14.10.2.4 Background Subtraction Method - Average of Front and Rear
14.10.2.5 Minimum percent for ID - 10% of nearest Internal Standard Area
14.10.2.6 Quantitation by - Area
14.10.2.7 RF Mode - Nearest ISTD

14.10.3 The library search does not use a normalization routine. These parameters are set in the Method and are not altered unless approved by a Supervisor or Manager. Note that the selection of peaks and the calculated amount uses the peak area of the nearest internal standard, not the response of quant ion. The area is found in the area.rp report in Target. The amount of an unknown is estimated using an RF factor of 1.

14.10.4 When performing a library search select Quant Unknown Compounds in Edit Quantitation Parameters and process the data file. The software will identify unknown peaks meeting the set criteria. If the software misses a peak that in the analyst opinion appears to meet the area requirements, the analyst may add an unknown. Select Spectra – Search, then Edit – Add Unknown. Select the TIC with the best fit (highest quality match). If no match is higher than 50% select the result as "Unknown". If in the analyst's opinion the Target selected best match is incorrect, the match is changed to reflect the best match or is reported as an unknown.

14.10.5 Review the assigned match using the following guidelines:

14.10.5.1 Major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

14.10.5.2 The relative intensities of the major ions should agree with the reference spectrum within ± 30%. Major ions that show increased intensity may be due to co-eluting compounds, and ions showing decreased intensity may be due to the background subtraction. When this is encountered examine the spectrum across the peak to determine if co-elution is an issue. If necessary, alter the background subtraction program to best handle the sample matrix.

14.10.5.3 Molecular ions present in reference spectrum should be present in sample spectrum (this is usually the heaviest ion).

14.10.6 Save changes and upload results to the LIMS. Only use the Quant Unknown Compound option when a library search is requested. If a library search is added after the file is processed, copy the file using the same file number and append a "T" behind the file number. Process the file and perform the TIC search. Do not reprocess a data file that has already been uploaded to the LIMS.

14.10.7 If a specific analyte is requested to report as a library search, perform the MS library search as described. If the analyte is identified, post the result. If the analyte is not identified, post the TIC file and enter the CAS

number, target analyte, and ND in the CAS/Name column of the sample results. Leave the amount column empty.

15.0 CALCULATIONS

15.1 % Recovery (MS/MSD) = $\frac{\text{Spike Concentration-Sample Concentration}}{\text{Spike Added}} \times 100$

15.2 % Recovery (LCS/LCSD/Surrogate) = $\frac{\text{Spike Concentration}}{\text{Spike Added}} \times 100$

15.3 RPD = $\frac{|\text{Concentration 2-Concentration 1}|}{(\text{Concentration 2+Concentration 1})/2} \times 100$

15.4 Response Factor $= A_s/C_s$

As= Peak Area of analyte or surrogate

Cs= Concentration of the analyte or surrogate

15.5 % Difference = $[(RF_I - RF_C) / RF_I] \times 100$

RF_I = Average response factor from initial calibration

RF_C = Response factor from current verification check standard

15.6 % RSD = (SD/X) X 100

RSD = Relative Standard Deviation

X = mean of initial RF's for a compound

SD = Standard Deviation of average RF's for a compound

15.7 Average Response Factor Calculation

Concentration (µg/L) = $\frac{(A_s \times DF)}{(RF)}$

Concentration (µg/kg) = $\frac{(A_s \times DF)}{(RF)}$

A_s= Response of the parameter to be measured excluding the surrogate and IS peaks.

RF= Average response factor from the initial calibration

DF= Dilution factor

16.0 REPORTING LIMIT / LIMIT OF QUANTITATION (LOQ)

16.1 Water samples – L3 calibration standard listed in Table 1A

16.2 Soil samples – L2 calibration standard listed in Table 1B

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in the analytical methods.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 20 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 10%. This shall be evaluated quarterly. Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the amount measured in any sample (whichever is greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately.

19.2 The LCS control limits specified in Table 3 are used to determine batch acceptability. If the full list of target analytes is spiked, a certain number of marginal sporadic failures is allowed. See Table 5 for criteria for marginal sporadic failures. If the LCS fails to meet the laboratory acceptance criteria, the source of inaccuracy should be investigated and samples should be reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.3 The MS control limits specified in Table 3 are used to determine sample matrix effect. If the recovery is outside of control limits and a lab error is suspected, repeat the MS determination. If the LCS is within control limits and matrix interference is indicated, report the results with a case narrative.

19.4 The LCS/LCSD and MS/MSD criteria are specified in Table 3. If the RPD is greater than the control limits in Table 3, investigate the source of the precision error. A source of precision error in the MS/MSD may be the non-homogenous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.5 The surrogate criteria are specified in Table 4. If a surrogate recovery is below QC limits in a MB, LCS, or LCSD, the entire batch must be re-analyzed. If a surrogate recovery is below QC limits in a sample, the sample must be reanalyzed. If the surrogate recovery is above limits then the data is considered acceptable if there are no target analytes detected in the associated samples.

19.6 Internal Standards (IS)

19.6.1 The criteria for internal standards for QC samples (ICV, CCV, MB, LCS, LCSD) is -50% to +100% of the daily CCV. QC samples may not be reported if IS criteria is not met.

19.6.2 The criteria for internal standards for investigative samples is -70% to +100% of the daily CCV. For samples with IS results outside criteria, determine if the IS failure is due to instrument malfunction or sample matrix. Corrective action if IS failure is due to instrument malfunction: rerun sample undiluted (or same dilution as original analysis). Corrective action if IS failure is due to sample matrix: run compounds that fail criteria at a 5x dilution, or use the methanol extract for soil samples.

19.6.3 For DoD projects, the criteria for internal standards for QC samples (ICV, CCV, MB, LCS, LCSD) and investigative samples is -50% to +100% of the ICAL midpoint.

19.6.4 The retention time of the IS in the sample must not shift greater than + 0.5 min of the retention time of the corresponding IS in the CCV.

19.7 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.8 Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative.

19.9 Spikes may be diluted out in the analysis process if a 10X or higher dilution is performed. Document this in the case narrative.

19.10 Project specific criteria may apply. See SOP GEN-019.

19.11 For data review procedures and checklists, see SOP QA-002.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 8000B
22.2 SW-846 8260B
22.3 SW-846 5030B
22.4 SW-846 5035

23.0 METHOD MODIFICATIONS

23.1 In cases where method 8260B does not specify acceptance criteria, GCAL uses internal criteria or project-specific criteria.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history
24.2 Table 1A: Water Calibration Concentrations
24.3 Table 1A: Soil Calibration Concentrations
24.4 Table 2: BFB Tuning Acceptance Criteria
24.5 Table 3: Control Limits for LCS/LCSD/MS/MSD and Internal Standard Associations
24.6 Table 4: Control Limits for Surrogates
24.7 Table 5: Criteria for Marginal Sporadic Failures

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
12/12/11	23	Previous version	
10/03/13	24	Updated format and added sections required for TNI-compliant SOPs. Incorporated information for oxygenates, previously documented in SOP GCMSV-004.	Shelley Bourgeois

Table 1A: Water Calibration Concentrations, (µg/L)

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	L9
Analyte concentration unless specified	0.2	0.4	1	5	10	20	50	100	200
Acrolein	1	2	5	25	50	100	250	500	1000
Acrylonitrile	1	2	5	25	50	100	250	500	1000
m,p-Xylene	0.4	0.8	2	10	20	40	100	200	400
Ethyl Ether	1	2	5	25	50	100	250	500	1000
Acetonitrile	1	2	5	25	50	100	250	500	1000
Ethyl Acetate	1	2	5	25	50	100	250	500	1000
Tetrahydrofuran	1	2	5	25	50	100	250	500	1000
Propionitrile	1	2	5	25	50	100	250	500	1000
Isobutyl Alcohol	1	2	5	25	50	100	250	500	1000
1,4-Dioxane	5	10	25	125	250	500	1250	2500	5000
Cyclohexanone	0.4	0.8	2	10	20	40	100	200	400
Benzal Chloride	0.4	0.8	2	10	20	40	100	200	400
Ethanol	N/A	N/A	504	2520	5040	10080	25200	50400	100800
Tert-amyl Alcohol	N/A	N/A	4	20	40	80	200	400	800
3,3-dimethyl-1-butanol	N/A	N/A	51	255	510	1020	2550	5100	10200

Note: Levels 1 and 2 are optional and may be removed from the calibration. The reporting limit is based on calibration Level 3.

Table 1B: Soil Calibration Concentrations, (µg/Kg)

Analyte	L1	L2	L3	L4	L5	L6	L7
Analyte concentration unless specified	2	5	10	20	50	100	200
Acrolein	10	25	50	100	250	500	1000
Acrylonitrile	10	25	50	100	250	500	1000
P,M-Xylene	8	10	20	40	100	200	400
Ethyl Ether	10	25	50	100	250	500	1000
Acetonitrile	10	25	50	100	250	500	1000
Ethyl Acetate	10	25	50	100	250	500	1000
Propionitrile	10	25	50	100	250	500	1000
Isobutyl Alcohol	10	25	50	100	250	500	1000
1,4-Dioxane	50	125	250	500	1250	2500	5000
Cyclohexanone	4	10	20	40	100	200	400

Table 2: BFB Acceptance Criteria

M/Z	ION ABUNDANCE CRITERIA
50	15 - 40% of Mass 95
75	30 - 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5 - 9% of Mass 95
173	Less than 2% of Mass 174
174	50 - 120 of Mass 95
175	5 - 9% of Mass 174
176	>95 but <101% of Mass 174
177	5 - 9% of Mass 176

Table 3: Control Limits for LCS/LCSD/MS/MSD and Internal Standard Associations

Analyte	Associated Internal Standard	Control Limits, Water (%)	Control Limits, Soil (%)	RPD Limit
1,1,1,2-Tetrachloroethane	2	75-124	77-122	30
1,1,1-Trichloroethane	1	76-126	70-130	30
1,1,2,2-Tetrachloroethane	3	70-122	66-129	30
1,1,2-Trichloroethane	2	72-121	74-120	30
1,1-Dichloroethane	1	74-127	71-126	30
1,1-Dichloroethene	1	69-129	68-129	30
1,1-Dichloropropene	1	72-131	70-138	30
1,2,3-Trichlorobenzene	3	65-131	69-130	30
1,2,3-Trichloropropane	3	70-120	63-132	30
1,2,4-Trichlorobenzene	3	61-135	64-135	30
1,2,4-Trimethylbenzene	3	74-125	75-130	30
1,2-Dibromo-3-chloropropane *	3	57-121	60-123	30
1,2-Dibromoethane	2	70-124	74-122	30
1,2-Dichlorobenzene	3	71-126	76-125	30
1,2-Dichloroethane	1	71-129	68-126	30
1,2-Dichloroethene (Total)	1	74-128	72-129	30
1,2-Dichloropropane	1	72-128	72-129	30
1,2-Diethylbenzene	3	60-140	70-130	30
1,3,5-Trimethylbenzene	3	71-132	74-136	30
1,3-Butadiene *	1	60-140	70-130	30
1,3-Dichlorobenzene	3	74-126	77-127	30
1,3-Dichloropropane	1	74-122	77-121	30
1,3-Dichloropropylene	1	72-131	73-128	30
1,3-Diethylbenzene	3	60-140	70-130	30
1,4-Dichloro-2-butene *	3	60-140	N/A	30

1,4-Dichlorobenzene	3	72-122	74-123	30
1,4-Diethylbenzene	3	60-140	70-130	30
1,4-Dioxane *	1	60-140	70-130	30
1-Bromo-2-Chloroethane	1	68-130	70-128	30
1-Chlorohexane	2	67-135	68-136	30
1-Nitropropane	2	60-140	50-150	30
2,2,4-Trimethylpentane *	1	60-140	N/A	30
2,2-Dichloropropane	1	77-124	74-129	30
2,3-Dichloro-1-propene	1	60-140	70-130	30
2-Butanone *	1	58-137	47-142	30
2-Chloroethylvinyl ether *	1	56-124	42-134	30
2-Chloropropene *	1	60-140	N/A	30
2-Chlorotoluene	3	72-127	75-132	30
2-Hexanone *	2	50-135	47-137	30
2-Nitropropane *	2	60-140	50-150	30
3,4-Dichloro-1-Butene	3	60-140	60-140	30
4-Chlorotoluene	3	75-126	74-133	30
4-Isopropyltoluene	3	71-129	71-136	30
4-Methyl-2-pentanone *	3	57-132	52-136	30
4-Methylstyrene	3	70-130	N/A	30
Acetone *	1	44-156	38-152	30
Acetonitrile *	1	60-140	70-130	30
Acrolein *	1	30-160	34-158	30
Acrylonitrile *	1	64-137	49-142	30
Allyl chloride	1	60-140	70-130	30
Alpha methyl styrene	3	60-140	N/A	30
Benzal chloride	3	60-140	70-130	30
Benzene	1	70-129	73-128	30
Benzyl chloride *	3	60-140	50-150	30
Bromobenzene	3	71-120	73-124	30
Bromochloromethane	1	76-130	73-127	30
Bromodichloromethane	1	74-125	74-126	30
Bromoform	2	64-122	67-122	30
Bromomethane	1	47-138	48-139	30
Carbon disulfide	1	69-136	68-133	30
Carbon tetrachloride	1	76-128	71-133	30
Chlorobenzene	2	74-123	75-121	30
Chloroethane	1	62-141	57-144	30
Chloroform	1	75-122	74-124	30
Chloromethane	1	59-132	61-130	30
Chloroprene	1	60-140	60-140	30
Cyclohexane	1	69-132	70-136	30

Cyclohexanone *	3	60-140	60-140	30
Dibromochloromethane	2	71-123	74-122	30
Dibromomethane	1	72-129	72-125	30
Dichlorodifluoromethane	1	58-140	59-138	30
Dicyclopentadiene *	3	60-140	N/A	30
Diethyl ether	1	60-140	70-130	30
Diethylbenzene (total)	3	60-140	70-130	30
Dimethyldisulfide	1	60-140	70-130	30
Ethanol *	1	70-130	60-140	30
Ethyl acetate *	1	60-140	70-130	30
Ethyl methacrylate *	2	60-140	70-130	30
Ethyl tert-butyl ether (ETBE) *	1	70-130	60-140	30
Ethylbenzene	2	74-126	74-130	30
Hexachlorobutadiene	3	61-144	71-140	30
Isobutyl alcohol *	1	60-140	50-150	30
Isopropylbenzene (Cumene)	2	71-125	74-125	30
Methacrylonitrile *	1	60-140	50-150	30
Methyl Acetate	1	57-139	49-138	30
Methyl iodide	1	57-141	54-140	30
Methyl methacrylate *	1	60-140	70-130	30
Methylcyclohexane	1	67-138	70-142	30
Methylene chloride	1	68-132	66-130	30
Naphthalene	3	57-138	54-132	30
Pentachloroethane *	3	60-140	N/A	30
Propionitrile *	1	60-140	70-130	30
Styrene	2	71-127	72-128	30
Tetrachloroethene	2	68-128	70-127	30
Tetrahydrofuran *	1	60-140	70-130	30
Toluene	2	72-120	74-121	30
Total Difluorobenzene	1	60-140	70-130	30
Trichloroethene	1	76-129	78-127	30
Trichlorofluoromethane	1	72-136	64-141	30
Trichlorotrifluoroethane	1	72-136	66-139	30
Vinyl acetate	1	54-147	53-140	30
Vinyl chloride	1	68-132	67-131	30
Xylene (total)	2	74-127	71-129	30
cis-1,2-Dichloroethene	1	73-130	72-130	30
cis-1,3-Dichloropropene	1	71-132	72-129	30
cis-1,4-Dichloro-2-Butene	3	60-140	60-140	30
Diisopropyl Ether (DIPE) *	1	70-130	70-130	30
m,p-Xylene	2	74-126	72-128	30
n-Butyl alcohol *	1	60-140	50-150	30

n-Butylbenzene	3	69-134	68-144	30
n-Heptane	1	40-160	40-160	30
n-Hexane	1	68-138	67-143	30
n-Propylbenzene	3	75-129	73-137	30
o-Xylene	2	73-130	69-133	30
sec-Butanol	1	60-140	60-140	30
sec-Butylbenzene	3	70-136	72-141	30
t-Butanol (TBA) *	1	60-140	60-140	30
t-amyl methyl ether (TAME) *	1	70-130	60-140	30
tert-Butyl methyl ether (MTBE)	1	71-125	69-126	30
tert-Butylbenzene	3	72-126	72-136	30
tert-amyl alcohol (TAA)	1	70-130	60-140	30
tert-butyl formate (TBF) *	1	70-130	N/A	30
trans-1,2-Dichloroethene	1	69-132	67-134	30
trans-1,3-Dichloropropene	1	71-131	72-126	30
trans-1,4-Dichloro-2-butene	3	56-132	44-146	30

* Analytes defined as poor performers

Internal Standards: 1 – Fluorobenzene
2 – Chlorobenzene-d5
3 – 1,4-Dichlorobenzene-d4

Table 4: Control Limits for Surrogate

Surrogate	Control Limits, Water (%)	Control Limits, Soil (%)
Toluene-d8	76-134	71-132
4-Bromofluorobenzene	78-130	62-127
1,2-Dichloroethane-d4	71-127	62-125
Dibromofluoromethane	77-127	65-130

Table 5: Marginal Sporadic Failures

Number of Analytes	Failures Allowed
<11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

STANDARD OPERATING PROCEDURE FOR

ACID DIGESTION OF SOLID SAMPLES FOR METALS ANALYSIS

SW-846 3050B

Approved by: _____ Date: 10/08/13
R. Jason Olivier, Metals Supervisor

Approved by: _____ Date: 10/08/13
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 10/08/13
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is designed for the preparation of sediment, sludge and soil samples for determination of total metals by ICP or ICP-MS.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Solid and Waste

3.0 SUMMARY OF TEST METHOD

3.1 For samples requiring ICP analysis, a representative sample is digested with repeated additions of nitric acid (HNO_3), hydrogen peroxide (H_2O_2), and hydrochloric acid (HCl). The digestate is then analyzed by method 6010C (SOP MET-010).

3.2 For samples requiring ICP-MS analysis, a representative sample is digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). The digestate is then analyzed by 6020A (SOP MET-021).

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.
7.4 Contact lenses are not to be worn in the laboratory.
7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 CPI Mod Block – Digestion Block
8.2 Hot plate
8.3 70 mL flip-top digestion vessels
8.4 250 mL glass digestion flasks
8.5 100 mL Specimen cups
8.6 Watch glasses
8.7 Teflon boiling beads (or equivalent)
8.8 Whatman 41 (or equivalent) filter paper
8.9 Eppendorf pipettes, various sizes, calibrated daily when in use
8.10 Balance accurate to 0.0001g and calibrated daily when in use
8.11 Water purification system, Millipore Synergy Ultrapure or equivalent system to produce Type 1 water (18.2 MΩ·cm at 25 °C ultrapure water)

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.
9.1.1 Type 1 reagent water
9.1.2 Nitric Acid – Trace Metal grade (ICP); Ultra trace metal grade (ICP-MS)
9.1.3 Hydrochloric Acid – Trace Metal grade (ICP); Ultra trace metal grade (ICP-MS)
9.1.4 Hydrogen Peroxide (30%)
9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all

standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock Standards (commercially prepared by Inorganic Ventures, multi-analyte mix, various concentrations)

9.2.1.1 Custom mix GCAL1A

9.2.1.2 Custom mix GCAL2C

9.2.1.3 Custom mix GCAL8

9.2.2 Stock Standards (commercially prepared, single analytes, 1000 mg/L each Antimony, Selenium, and Silver)

9.2.3 Sb/Se/Ag Working Standard (20/2/20 mg/L) – Add 500 µL of antimony stock standard, 100 µL of selenium stock standard, and 500 µL of silver stock standard to a 50 mL volumetric flask and dilute to volume with 2% nitric acid.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – none

11.2 Maximum holding time – 6 months from collection to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to blank glass beads or Teflon chips and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike (MS) shall be analyzed per twenty samples. The MS is a known amount of standard added to a sample at the same concentration as the LCS.

12.4 One laboratory duplicate (DUP) and/or matrix spike duplicate (MSD) shall be analyzed per twenty samples.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified daily. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook.

14.0 PROCEDURE

14.1 Mod Block Digestion

14.1.1 Label a 70 mL digestion vessel with the sample ID for each sample to be digested. Fill in the metals prep sheet as each step is completed.

14.1.2 With each batch, a method blank, LCS, and MS/MSD or MS/DUP are included. If insufficient sample is available for an MSD or DUP, document on the prep sheet and prepare an LCSD with the batch.

14.1.3 Weigh 1.25 g – 1.30 g of Teflon chips into the vessel for the blank and LCS. Record the weight on the prep sheet.

14.1.4 Homogenize each of the wet samples by mixing well. The sample may be removed from the container and placed on butcher paper to allow for mixture of the whole sample. Weigh 1.25 g – 1.30 g of a homogenized sample into a labeled 70 mL digestion vessel. Record the weight to the nearest 0.01g.

14.1.5 Add spiking solutions to the LCS, MS, and MSD vessels as described below:

14.1.5.1 For ICP analysis, add 250 µL of GCAL1A and 250 µL of GCAL2C.

14.1.5.2 For ICP-MS analysis, add 250 µL of GCAL8 and 250 µL of Sb/Se/Ag spike.

14.1.6 Add 5 mL of 1:1 HNO_3 to each vessel and mix.

14.1.7 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.1.8 Place the digestion vessels in the block, cover with a watch glass and reflux the samples for 15 minutes without boiling.

14.1.9 Cool and add 2.5 mL of concentrated HNO_3 to each sample; replace the watch glasses and reflux for 30 minutes.

14.1.10 If brown fumes are generated, indicating oxidation of the sample by the HNO_3, repeat step 14.1.9 until no brown fumes are given off by the sample, indicating the reaction is complete.

14.1.11 Cool. Add 1 mL Type 1 water and 1.5 mL 30% of H_2O_2 (peroxide) and cover with the watch glass.

14.1.12 On low heat, warm the sample to start the H_2O_2 reaction and heat until any effervescence subsides. Continue adding 30% H_2O_2 in 1 mL aliquots

(up to a maximum total of 5 mL of 30% H_2O_2) until the reaction is minimal.

14.1.13 Cover the sample with the watch glass and heat at 90-95°C for 2 hours or until the sample volume is approximately 5 mL.

14.1.14 For samples requiring ICP analysis only, add 5 mL of concentrated HCl, cover with a watch glass and reflux at 90-95°C for 15 minutes.

14.1.15 Cool and adjust final volume to 50 mL with Type 1 water.

14.1.16 Filter the digestate through a Whatman 41 (or equivalent) filter; this step may be done in the laboratory prior to analysis.

14.2 Hot Plate Digestion – this procedure is used for waste/liquid organic samples

14.2.1 Homogenize the sample by mixing well. Weigh 1.25 g – 1.30 g of a homogenized sample into a labeled 250 mL glass digestion flask. Record the weight to the nearest 0.01g. Prepare QC samples as required in the quality control section.

14.2.2 Add 5 mL of 1:1 HNO_3 and mix.

14.2.3 Set the hot plate to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.2.4 Place the digestion flasks on the hot plate, cover with a watch glass and reflux the samples for 15 minutes without boiling.

14.2.5 Cool and add 2.5 mL of concentrated HNO_3 to each sample; replace the watch glasses and reflux for 30 minutes.

14.2.6 If brown fumes are generated, indicating oxidation of the sample by the HNO_3, repeat step 14.2.5 until no brown fumes are given off by the sample, indicating the reaction is complete. For concentrated waste samples, this step is performed many times and therefore the digestion may take several days.

14.2.7 Cool. Add 1 mL Type 1 water and 1.5 mL 30% of H_2O_2 (peroxide) and cover with the watch glass.

14.2.8 On low heat, warm the sample to start the H_2O_2 reaction and heat until any effervescence subsides. Continue adding 30% H_2O_2 in 1 mL aliquots (up to a maximum total of 5 mL of 30% H_2O_2) until the reaction is minimal.

14.2.9 Cover the sample with the watch glass and heat at 90-95°C for 2 hours or until the sample volume is approximately 5 mL.

14.2.10 For samples requiring ICP analysis only, add 5 mL of concentrated HCl, cover with a watch glass and reflux at 90-95°C for 15 minutes.

14.2.11 Cool and adjust final volume to 50 mL with Type 1 water.

14.2.12 Filter the digestate through a Whatman 41 (or equivalent) filter; this step may be done in the laboratory prior to analysis.

14.3 Alternate Prep for Antimony

14.3.1 Homogenize the wet sample by mixing well. The sample may be removed from the container and placed on butcher paper to allow for mixture of the whole sample. Weigh 2.50 – 2.60 g of a homogenized

sample into a labeled 70 mL digestion vessel. Record the weight to the nearest 0.01g. Prepare QC samples as required in the quality control section.

14.3.2 Add 3 mL of concentrated HNO_3 and 10 mL concentrated HCl and mix.

14.3.3 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.3.4 Place the digestion vessels in the block, cover with a watch glass and reflux the samples for 15 minutes without boiling.

14.3.5 Cool and filter the digestate through a Whatman 41 (or equivalent) filter. Collect the filtrate in another labeled 70 mL digestion vessel. Rinse the filter paper with no more than 5.0 mL of hot HCl (~95°C), then with 20 mL of hot reagent water (~95°C). Collect washings in same digestion vessel as the filtrate.

14.3.6 Remove the filter and residue from funnel and place in original digestion vessel. Add 5.0 mL concentrated HCl, place vessel back in block with watch glass and wash the sides of digestion vessel with reagent water. Repeat step 14.3.5 filter process.

14.3.7 Cool, transfer the digestate to a 100 mL specimen cup, and adjust the final volume to 100 mL with Type 1 water. If precipitation occurs in digestion tube upon cooling the primary or secondary filtrate, do not bring up to volume. Instead, add up to 10 mL of concentrated HCl to dissolve precipitate. Once precipitate is dissolved, bring to volume with reagent water.

14.4 Glassware Cleaning – all vessels, watch glasses, and volumetric measuring devices used in metals prep are disposable and therefore do not require cleaning, with the exception of the 250 mL glass digestion flasks used to perform the hot plate digestion in Procedure 14.2.

14.4.1 Thoroughly wash each glass flask with detergent and tap water. This may be performed by hand or in the laboratory dishwasher.

14.4.2 Rinse the glassware with DI water and then with 1:1 HNO_3.

14.4.3 Follow the acid rinse with another DI water rinse.

14.4.4 Air dry.

15.0 CALCULATIONS

15.1 Not applicable

16.0 REPORTING LIMITS

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs MET-010 and MET-021 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 3050B

23.0 METHOD MODIFICATIONS

23.1 The final volume has been adjusted from 100 mL to 50 mL. Ratios of reagents added have been adjusted accordingly.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep Sheets are located on the GCAL Intranet Main Menu, under Laboratory, Prep Menu, Metals Forms

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
10/01/11	14	Previous version	James Turner
07/27/12	15	Revised to meet method requirements.	Abigail Guerin
10/08/13	16	Updated format and added sections required for TNI-compliant SOPs. Added the purification unit for Type 1 water. Corrected the prep procedure for ICP-MS. Added the custom spiking solutions. Incorporated the glassware cleaning procedure, previously in SOP MET-001.	Shelley Bourgeois

STANDARD OPERATING PROCEDURE FOR

ACID DIGESTION OF AQUEOUS SAMPLES FOR METALS ANALYSIS

SW-846 3010A

Approved by: _____ Date: 10/08/13
R. Jason Olivier, Metals Supervisor

Approved by: _____ Date: 10/08/13
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 10/08/13
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is designed for the preparation of aqueous samples and extracts for determination of total metals by ICP or ICP-MS.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Aqueous samples and TCLP/SPLP extracts

3.0 SUMMARY OF TEST METHOD

3.1 For samples requiring ICP analysis, a representative sample is digested with repeated additions of nitric acid (HNO_3) and hydrochloric acid (HCl). The digestate is then analyzed by method 6010C (SOP MET-010).

3.2 For samples requiring ICP-MS analysis, a representative sample is digested with repeated additions of nitric acid (HNO_3) and hydrochloric acid (HCl). The digestate is then analyzed by 6020A (SOP MET-021).

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Interferences are discussed in the analytical SOPs.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye

covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 CPI Mod Block – Digestion Block

8.2 70 mL flip-top digestion vessels

8.3 Watch glasses

8.4 Nalgene 50mm filter units with 0.45um filters

8.5 Eppendorf pipettes, various sizes, calibrated daily when in use

8.6 Balance accurate to 0.0001g and calibrated daily when in use

8.7 Water purification system, Millipore Synergy Ultrapure or equivalent system to produce Type 1 water (18.2 MΩ·cm at 25 °C ultrapure water)

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Type 1 reagent water

9.1.2 Nitric Acid – Trace Metal grade (ICP); Ultra trace metal grade (ICP-MS)

9.1.3 Hydrochloric Acid – Trace Metal grade (ICP); Ultra trace metal grade (ICP-MS)

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock Standards (commercially prepared by Inorganic Ventures, multi-analyte mix, various concentrations)

9.2.1.1 Custom mix GCAL1A

9.2.1.2 Custom mix GCAL2C

9.2.1.3 Custom mix GCAL8

9.2.2 Stock Standards (commercially prepared, single analytes, 1000 mg/L each Antimony, Selenium, and Silver)

9.2.3 Sb/Se/Ag Working Standard (20/2/20 mg/L) – Add 500 µL of antimony stock standard, 100 µL of selenium stock standard, and 500 µL of silver stock standard to a 50 mL volumetric flask and dilute to volume with 2% nitric acid.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation

11.1.1 Total metals – HNO_3 to pH <2

11.1.2 Dissolved metals – filter through a 0.45 micron filter on site, then HNO_3 to pH <2

11.2 Maximum holding time – 6 months from collection to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to DI water and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike (MS) shall be analyzed per twenty samples. The MS is a known amount of standard added to a sample at the same concentration as the LCS.

12.4 One laboratory duplicate (DUP) and/or matrix spike duplicate (MSD) shall be analyzed per twenty samples.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Auto-Pipettes will be calibrated each quarter and verified daily. Quarterly calibrations of auto-pipettes are recorded in the Non-Standard Labware Logbook and daily calibration verifications for auto- pipettes are recorded in the Daily Mechanical Pipet Calibration logbook.

14.0 PROCEDURE

14.1 Total Metals and Extracts

14.1.1 Label a 70 mL digestion vessel with the sample ID for each sample to be digested. Fill in the metals prep sheet as each step is completed.

14.1.2 With each batch, a method blank, LCS, and MS/MSD or MS/DUP are included. If insufficient sample is available for an MSD or DUP, document on the prep sheet and prepare an LCSD with the batch.

14.1.3 Measure 50 mL of Type 1 water for the blank and LCS. Record the volume on the prep sheet.

14.1.4 Homogenize each of the samples by mixing well. Measure 50 mL of well-mixed sample into a labeled 70 mL digestion vessel. Record the volume on the prep sheet.

14.1.5 Add spiking solutions to the LCS, MS, and MSD vessels as described below:

14.1.5.1 For ICP analysis, add 250 µL of GCAL1A and 250 µL of GCAL2C.

14.1.5.2 For ICP-MS analysis, add 250 µL of GCAL8 and 250 µL of Sb/Se/Ag spike.

14.1.6 Add 1.5 mL of concentrated HNO_3 to each vessel and mix.

14.1.7 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.1.8 Place the digestion vessels in the block, cover with a watch glass and heat the samples at 90-95°C for 2 hours or until the sample volume reaches approximately 5 mL. Do not allow the sample to boil.

14.1.9 Cool and add 1.5 mL of concentrated HNO_3 to each sample; replace the watch glasses and reflux.

14.1.10 If brown fumes are generated, indicating oxidation of the sample by the HNO_3, repeat step 14.1.9 until no brown fumes are given off by the sample, indicating the reaction is complete.

14.1.11 Remove samples from the digestion block and allow to cool.

14.1.12 For samples requiring ICP analysis only, add 5 mL of 1:1 HCl, cover with a watch glass and reflux at 90-95°C for 15 minutes.

14.1.13 For samples requiring ICP-MS analysis only, add 0.5 mL of 1:1 HCl, cover with a watch glass and reflux at 90-95°C for 15 minutes.

14.1.14 Cool and adjust final volume to 50 mL with Type 1 water.

14.2 Dissolved Metals (field-filtered)

14.2.1 When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

14.2.2 Prepare samples as described in Procedure steps 14.1.1 – 14.1.5 and deliver to the metals laboratory.

14.3 Dissolved Metals (not field-filtered)

14.3.1 Filter an aliquot of Type 1 water and all samples for analysis through a 0.45 micron filter.

14.3.2 Preserve the filtrate with 1 mL concentrated HNO3 and hold for 24 hours.

14.3.3 Prepare samples as described in Procedure steps 14.1.1 – 14.1.5 and deliver to the metals laboratory.

15.0 CALCULATIONS

15.1 Not applicable

16.0 REPORTING LIMITS

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOPs MET-010 and MET-021 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SW-846 3010A

23.0 METHOD MODIFICATIONS

23.1 The final volume has been adjusted from 100 mL to 50 mL. Ratios of reagents added have been adjusted accordingly.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Prep Sheets are located on the GCAL Intranet Main Menu, under Laboratory, Prep Menu, Metals Forms

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
10/01/11	14	Previous version	James Turner
07/27/12	15	Revised to meet method requirements and for clarity.	Abigail Guerin
10/08/13	16	Updated format and added sections required for TNI-compliant SOPs. Added the purification unit for Type 1 water. Corrected the prep procedure for ICP-MS. Added the custom spiking solutions.	Shelley Bourgeois

STANDARD OPERATING PROCEDURE FOR

ACID DIGESTION OF AQUEOUS AND SOLID SAMPLES FOR MERCURY ANALYSIS

EPA 245.2 / EPA 7470A / EPA 7471B

Approved by: _____ Date: 12/05/13
R. Jason Olivier, Inorganics Manager

Approved by: _____ Date: 12/05/13
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 12/05/13
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is designed for the preparation of aqueous samples, TCLP/SPLP extracts, sediment, sludge, and soil samples for determination of total or dissolved mercury by FIMS analysis.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Aqueous, Solid, and Waste

3.0 SUMMARY OF TEST METHOD

3.1 Prior to analysis, samples must be prepared according to the procedure discussed in this SOP. Following acid digestion, samples are analyzed using the appropriate procedure (see GCAL SOP MET-008).

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Interferences are discussed in the analytical SOP (see GCAL SOP MET-008).

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.
7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 CPI Mod Block – Digestion Block
8.2 70 mL flip-top digestion vessels
8.3 100 mL Specimen cups
8.4 Metals Free (Blank) Sand
8.5 Whatman 41 (or equivalent) filter paper
8.6 Nalgene 50mm filter units with 0.45um filters
8.7 Eppendorf pipettes, various sizes, calibrated daily when in use
8.8 Repipette Bottle Dispenser, calibrated quarterly
8.9 Balance accurate to 0.0001g and calibrated daily when in use
8.10 Water purification system, Millipore Synergy Ultrapure or equivalent system to produce Type 1 water (18.2 MΩ·cm at 25 °C ultrapure water)
8.11 Mechanical stir plate and stir bar
8.12 1000 mL beaker
8.13 200 mL, 1000 mL, and 2000 mL volumetric flasks

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized (DI) water
9.1.2 Nitric Acid (HNO_3) – Trace Metal grade
9.1.3 Hydrochloric Acid (HCl) – Trace Metal grade
9.1.4 Sulfuric Acid (H_2SO_4) –Trace Metal grade
9.1.5 2% HNO3 – In a 1 L volumetric flask, add 20 mL of concentrated HNO3 and dilute to volume with DI water.
9.1.6 Aqua Regia – In a 1000 mL Beaker, carefully combine 600 mL of concentrated HCl and 200 mL of concentrated HNO_3. Mix well on a mechanical stir plate with stir bar.
9.1.7 5% Potassium Permanganate – In a 2L volumetric flask, add 100 g of Potassium Permanganate and dilute to volume with DI water. Mix well on a mechanical stir plate with stir bar.

9.1.8 5% Potassium Persulfate – In a 2L volumetric flask, add 100 g of Potassium Persulfate and dilute to volume with DI water. Mix well on a mechanical stir plate with stir bar.

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock CCV Standard (commercially prepared, 1000 mg/L Mercury)

9.2.2 Stock ICV standard (commercially prepared, 1000 mg/L Mercury)

9.2.3 Working CCV Standard (0.1 mg/L Mercury) – Add 20 µL of the CCV Mercury stock standard to a 200 mL volumetric flask. Dilute to volume with 2% HNO_3. Working standards expire 28 days from date prepared or manufacturer's expiration date, whichever comes first. Standard may be prepared more frequently as needed. This standard is made by the metals laboratory.

9.2.4 Working ICV Standard (0.1 mg/L Mercury) – Add 20 µL of the ICV Mercury stock standard to a 200 mL volumetric flask. Dilute to volume with 2% HNO_3. Working standards expire 28 days from date prepared or manufacturer's expiration date, whichever comes first. Standard may be prepared more frequently as needed. This standard is made by the metals laboratory.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation

11.1.1 Aqueous Samples

11.1.1.1 Total mercury – HNO_3 to pH <2

11.1.1.2 Dissolved mercury – filter through a 0.45 micron filter on site, then HNO_3 to pH <2

11.1.2 Solid and Waste samples – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 28 days from collection to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is an amount of blank sand prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to blank sand and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike (MS) shall be analyzed per twenty samples. The MS is a known amount of standard added to a sample at the same concentration as the LCS.

12.4 One laboratory duplicate (DUP) and/or matrix spike duplicate (MSD) shall be analyzed per twenty samples.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Analytical balances will be calibrated each day in use and the calibration recorded in the logbook associated with that analytical balance.

13.2 Mechanical pipettes will be calibrated each quarter and verified each day of use. Quarterly calibrations are recorded in the Volumetric Labware Verification Logbook and daily pipette verifications are recorded in the Daily Mechanical Pipet Calibration logbook.

14.0 PROCEDURE

14.1 Aqueous Samples (Waters/TCLP Extracts/SPLP Extracts)

14.1.1 Label a 70 mL digestion vessel with the sample ID and test for each sample to be digested. Fill in the metals prep sheet as each step is completed.

14.1.2 With each batch, a MB, LCS, and MS/MSD or MS/DUP are included. If insufficient sample is available for an MSD or DUP, document on the prep sheet and prepare an LCSD with the batch.

14.1.3 Measure 20 mL of DI water for the blank and LCS. Record the volume on the prep sheet.

14.1.4 Homogenize each of the samples by mixing well. Measure 20 mL of well-mixed sample into a labeled 70 mL digestion vessel. Record the volume on the prep sheet.

14.1.5 Add 1 mL of the 0.1 mg/L working CCV spiking solution to the LCS, MS, and MSD.

14.1.6 Add 1.0 mL of H_2SO_4 and 0.5 mL concentrated HNO_3 mixing after each addition.

14.1.7 Add 3 mL of 5% Potassium Permanganate solution. Shake and let stand for 15 minutes to ensure the purple color persists. Add additional Potassium Permanganate solution if necessary and record additional volume on prep log.

14.1.8 Add 1.6 mL Potassium Persulfate solution.

14.1.9 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.1.10 Close the lid on the digestion vessels and make a small slit in each lid using a box cutter. Place the vessels in the Mod Block and heat at 90-95°C for 2 hours.

14.1.11 Cool and deliver samples to the metals laboratory for analysis.

14.2 Solid Samples (Sediments/Sludges/Soils)

14.2.1 Label a 70 mL digestion vessel with the sample ID and test for each sample to be digested. Fill in the metals prep sheet as each step is completed.

14.2.2 With each batch, a MB, LCS, and MS/MSD or MS/DUP are included. If insufficient sample is available for an MSD or DUP, document on the prep sheet and prepare an LCSD with the batch.

14.2.3 Weigh 0.5 g – 0.6 g of blank sand into the vessel for the MB and LCS. Record the weight on the prep sheet.

14.2.4 Homogenize each of the wet samples by mixing well. The sample may be removed from the container and placed on butcher paper to allow for mixture of the whole sample. Weigh 0.5 g – 0.6 g of a homogenized sample into a labeled 70 mL digestion vessel. Record the weight to the nearest 0.01g.

14.2.5 Add 1.5 mL of the 0.1 mg/L working CCV spiking solution to the LCS, MS, and MSD.

14.2.6 Add 5 mL of DI water and add 5 mL of Aqua Regia.

14.2.7 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.2.8 Close the lid on the digestion vessels and make a small slit in each lid using a box cutter. Place the vessels in the Mod Block and heat at 90-95°C for 2 minutes.

14.2.9 Cool and add 30 mL DI water and 15 mL 5% Potassium Permanganate solution, and mix thoroughly. Add additional Potassium Permanganate as needed to maintain purple color. Record additional volume added on prep sheet.

14.2.10 Place the vessels in the Mod Block and heat at 90-95°C for 30 minutes.

14.2.11 Cool and deliver samples to the metals laboratory for analysis.

14.3 Liquid Organic Samples

14.3.1 Label a 70 mL digestion vessel with the sample ID and test for each sample to be digested. Fill in the metals prep sheet as each step is completed.

14.3.2 With each batch, a MB, LCS, and MS/MSD or MS/DUP are included. If insufficient sample is available for an MSD or DUP, document on the prep sheet and prepare an LCSD with the batch.

14.3.3 Weigh 0.5 g – 0.6 g of blank sand into the vessel for the MB and LCS. Record the weight on the prep sheet.

14.3.4 Homogenize each of the wet samples by mixing well. The sample may be removed from the container and placed on butcher paper to allow for mixture of the whole sample. Weigh 0.5 g – 0.6 g of a homogenized sample into a labeled 70 mL digestion vessel. Record the weight to the nearest 0.01g.

14.3.5 Add 1.5 mL of the 0.1 mg/L working CCV spiking solution to the LCS, MS, and MSD.

14.3.6 Add 5 mL of concentrated HNO_3 and 5 mL of concentrated HCl. Cap digestion tube and use a small knife to cut a slit in the cap.

14.3.7 Heat in the Mod Block until the fumes are clear or unchanging. Additional acid may be added to complete the digestion.

14.3.8 Add 30 mL of deionized water.

14.3.9 Add 15 mL Potassium Permanganate solution and mix thoroughly.

14.3.10 Set Mod Block to the required setting for temperature of 90-95°C. Allow 15 minutes for the block to stabilize at the appropriate temperature.

14.3.11 Place the vessels in the Mod Block and heat at 90-95°C for 30 minutes.

14.3.12 Cool and deliver samples to the metals laboratory for analysis.

14.4 Calibration Standards

14.4.1 Calibration standards are prepared at the levels below and processed through the sample preparation procedure for waters or soils as applicable. The standards are prepared at levels of 0.2 µg/L, 0.5 µg/L, 2 µg/L, 5 µg/L, and 10 µg/L, as well as a calibration blank and an ICV at 5 µg/L. Calibration standards are prepared using the Working CCV and ICV Spiking solutions. A calibration curve must be prepared daily when samples are being digested.

15.0 CALCULATIONS

15.1 Not applicable

16.0 REPORTING LIMITS

16.1 Not applicable. The reporting limit will be established by the analytical procedure.

17.0 METHOD PERFORMANCE

17.1 Refer to the determinative methods for performance data.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.
18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 Refer to the determinative methods and SOP MET-008 for data assessment and acceptance criteria.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:
20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).
20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 245.2
22.2 SW-846 7470A
22.3 SW-846 7471B

23.0 METHOD MODIFICATIONS

23.1 The volume prepared for aqueous samples has been adjusted from 100 mL to 20 mL. Ratios of reagents added have been adjusted accordingly.
23.2 Adjusted the amount of water added during for the solid prep procedure.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history
24.2 Prep Sheets are located on the GCAL Intranet Main Menu, under Laboratory, Prep Menu, Metals Forms

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
05/19/10	19	Previous version	James Turner
12/05/13	20	Updated format and added sections required for TNI-compliant SOPs. Added the purification unit for Type 1 water. Updated amount of potassium persulfate used in water prep to match method requirements.	Shelley Bourgeois

STANDARD OPERATING PROCEDURE FOR

ANALYSIS OF AQUEOUS AND SOLID SAMPLES FOR MERCURY

EPA 245.2 / EPA 7470A / EPA 7471B

Approved by: _____ Date: 12/05/13
R. Jason Olivier, Inorganic Manager

Approved by: _____ Date: 12/05/13
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 12/05/13
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This procedure is used for the determination of dissolved or total mercury in aqueous samples, TCLP/SPLP extracts, sediment, sludge, and soil samples by the cold vapor atomic absorption (CVAA) technique. A digestion procedure is required for all samples and standards.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Aqueous, Solid, and Waste

3.0 SUMMARY OF TEST METHOD

3.1 Prior to analysis, samples should be digested using the appropriate sample preparation methods (see GCAL SOP MET-006).

3.2 A prepared sample enters the system and is mixed with a reducing agent (stannous chloride) to form elemental mercury vapor. The mixture flows into a liquid-gas separator where argon is introduced to carry the Mercury vapor through the PTFE-Membrane Filter for water vapor removal. The dry vapor enters the optical cell. The mercury lamp delivers a stable source of emission at 253.7 nm. Absorbance by the mercury cold vapor is measured using a photocell detector with a wide dynamic range.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC) as described in SOP GEN-007.

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Interferences from concentrations as high as 20 ppm sulfide are eliminated by adding potassium permanganate.

6.2 Copper can interfere at concentrations above 10 ppm.

6.3 High chloride concentrations in seawaters, brines, and industrial effluents may require additional permanganate. During the oxidation step, chloride is converted into free chlorine which absorbs radiation at 253.7 nm.

6.4 Certain volatile organic materials absorb at 253.7 nm. Additional permanganate may help to reduce the interference. A smaller sample size may be digested.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Perkin Elmer FIMS 400 Mercury Analyzer with computer and printer

8.2 Perkin Elmer AS 90 auto–sampler

8.3 Fias Pump tubing of various sizes

8.4 50 mL centrifuge tubes

8.5 15 mL centrifuge vials

8.6 200 mL, 1000L, and 2000 mL volumetric flasks

8.7 Eppendorf pipettes, various sizes, calibrated daily when in use

8.8 Re-pipette Bottle Dispenser, calibrated quarterly

8.9 Water purification system, Millipore Synergy Ultrapure or equivalent system to produce Type 1 water (18.2 MΩ·cm at 25 °C ultrapure water)

8.10 Mechanical Stir Plate with stir bar

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Rinse bath - Approximately 10% HCl. In a 1000 mL Nalgene bottle, add 100 mL of concentrated trace metals grade HCl. Dilute to volume with

purified DI water. Expiration date will be 1 month from date prepared or manufacturer's expiration date whichever occurs first.

9.1.2 Stannous chloride – In a 2 L volumetric flask add 20 g stannous chloride and 100mL of concentrated trace metals grade HCl. Dilute to volume with purified DI water for a 1% solution. Mix well on a mechanical stir plate with stir bar to dissolve the stannous chloride. Prepare fresh daily.

Note: 1% Stannous Chloride Solution recommended for Perkin Elmer for FIMS 400 operation.

9.1.3 Sodium chloride – hydroxylamine sulfate solution: In a 1 L volumetric flask, dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in purified DI water and dilute to 1 liter. Mix well on a mechanical stir plate with stir bar. Expiration date will be 1 month from the date prepared or manufacturer's expiration date whichever occurs first.

9.1.4 3% HCl solution for carrier – In a 2L volumetric flask, add 60 mL of concentrated trace metals grade HCl and dilute to volume with purified DI water. The expiration date will be 1 month from date prepared or manufacturer's expiration date whichever occurs first.

Note: 3% HCl Solution recommended for Perkin Elmer for FIMS 400 operation.

9.1.5 2% HNO3 – In a 1L volumetric flask, add 20mL of concentrated trace metals grade HNO3 and dilute to volume with purified DI water. Expiration date will be 1 month from date prepared or manufacturer's expiration date whichever occurs first.

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Mercury – 1000 mg/L stock standard (commercially prepared)

9.2.2 Mercury ICV – 1000 mg/L stock standard (commercially prepared)

9.2.3 Working Standard (0.1 mg/L) – Add 20 µL of the Mercury stock standard to a 200 mL volumetric flask. Dilute to volume with 2% HNO_3. Working standards expire 28 days from date prepared or manufacturer's expiration date, whichever comes first. Standard may be prepared more frequently as needed.

9.2.4 ICV Working Standard (0.1 mg/L) – Add 20 µL of the ICV Mercury stock standard to a 200 mL volumetric flask. Dilute to volume with 2% HNO_3. Working standards expire 28 days from date prepared or manufacturer's expiration date, whichever comes first. Standard may be prepared more frequently as needed.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation

11.1.1 Aqueous Samples

11.1.1.1 Total mercury – HNO_3 to pH <2

11.1.1.2 Dissolved mercury – filter through a 0.45 micron filter on site, then HNO_3 to pH <2

11.1.2 Solid and Waste samples – Cool to 0-6° C, but not frozen

11.2 Maximum holding time – 28 days from collection to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to blank matrix and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike (MS) shall be analyzed per twenty samples. The MS is a known amount of standard added to a sample at the same concentration as the LCS.

12.4 One laboratory duplicate (DUP) and/or matrix spike duplicate (MSD) shall be analyzed per twenty samples.

12.5 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Initial Calibration (ICAL) – The initial calibration consists of a calibration blank and a minimum of five contiguous non-zero points. Additional points may be analyzed to better define the linear range and the lower limit of quantitation. The low or high points may be removed from a multi-point curve as long as a minimum of five points remain. The curve must exhibit a correlation coefficient (r) of ≥ 0.995 to be considered acceptable. A successful initial calibration curve must be analyzed before any result can be quantitated. A new curve must be performed at a minimum daily.

13.2 Initial Calibration Verification (ICV) – Immediately following the initial calibration and prior to sample analysis, the calibration is verified with a mid-level ICV standard. The acceptance criteria is 90-110% .The ICV standard must be prepared from a second source standard at or near the mid-range of the calibration curve. If the criteria referenced above are not achieved, corrective action must be taken. This may include re-analysis of the ICV or initial calibration, instrument maintenance, or re-preparation and reanalysis of the standards involved.

13.3 Initial Calibration Blank (ICB) – The ICB is analyzed at the beginning of the analysis prior to sample analysis. The ICB must be less than the LOQ, and for DOD projects, the ICB must be less than the LOD. A failure above the reporting limit may be reported with a narrative as long as the associated samples are either non-detect, or the failure is less than 10 times the concentration in the associated samples.

13.4 Continuing Calibration Verification (CCV) – The CCV is analyzed after every 10 samples (or more frequently) and at the end of analysis. The recovery must be 90-110%. If a CCV is outside control limits, the previous ten samples must be re-analyzed. High failures in the CCV may be reported with a narrative if the associated samples are non-detect.

13.5 Continuing Calibration Blank (CCB) – The CCB is analyzed after every 10 samples (or more frequently) and at the end of analysis. The CCB must be less than the LOQ, and for DOD projects, the CCB must be less than the LOD. A failure above the reporting limit may be reported with a narrative as long as the associated samples are either non-detect, or the failure is less than 10 times the concentration in the associated samples.

13.6 Samples reported shall be diluted for mercury greater than the high level calibration standard. Dilute so that the expected concentration is approximately in the mid-level of the curve.

13.7 Determine DLs annually using the requirements of SOP QA-009. DLs will be determined more frequently if there is a change to the method or instrumentation that may alter the sensitivity of the measurement. DLs should be less than ½ the reporting limit.

14.0 PROCEDURE

14.1 Instrument Operation and Sample Analysis

14.1.1 Open the FIMS 400 software by double clicking on the WINLAB32 icon. Turn the Hg lamp on. Allow the lamp to warm up for at least 30 minutes.

14.1.2 Replace the sample reductant, carrier, and waste tubing daily or as needed when in use.

14.1.3 Wash and acid rinse the rinse bath and fill with 10% HCl as needed.

14.1.4 Place reductant uptake tubing into the container of stannous chloride.

14.1.5 Place the carrier uptake tubing into the container of 3% HCl.

14.1.6 Replace the membrane filter on the liquid gas separator daily when in use.

14.1.7 Adjust the argon source pressure to 60 psi and confirm on the Argon flowmeter on the front panel of the instrument.

14.1.8 Prepare a cover page – record the laboratory ID and expiration date for the calibration standard, ICV, stannous chloride, sodium chloride-hydroxylamine solution, carrier acid, and the Hg acid rinse.

14.1.9 Prepare a sequence log from the LIMS system/Filemaker Pro program.

14.1.10 Prepare digested standards and samples for analysis by adding sodium chloride–hydroxylamine sulfate solution to reduce the excess permanganate (decolorize). For waters add 3 mL and for solids add 6 mL.

14.1.11 Place calibration standards in the standards rack. Standards and their positions are: (blank) – 1; 0.2 µg/L – 2; 0.5 µg/L – 3; 2.0 µg/L – 4; 5.0 µg/L – 5; 10.0 µg/L – 6.

14.1.12 Fill sample tubes with the following quality control samples:

Position	Standard
9	5 µg/L – Independent Source (ICV)
1	0.0 (ICB)
5	CCV
1	CCB

14.1.13 Fill sample tubes with approximately 5mL of sample and place in the position in the rack corresponding to their positions on the sequence log.

14.1.14 To type in sample descriptions for analysis, bring up the sample information file. Type in the sample ID next to the corresponding auto-sampler location, weight, volume, and dilution information. In the remarks field enter instrument Id, analyst and batch number. Auto-sampler positions available for the Perkin Elmer AS-90 are 17 to 106. Save the sample information file, and then close this window.

14.1.15 Open the automated analysis window and select the mercury method to be used. Type in the auto–sampler locations to be analyzed and select the result data set to be used.

14.1.16 Select the analyze tab at the bottom of the automated analysis window. Click on analyze all to calibrate and then run samples.

14.1.17 See section 13.0 or 19.0 for all initial calibration and analysis QC requirements.

14.1.18 Allow the instrument to flush the lines for a few minutes after the last sample is completed. Place the stannous chloride tubing in purified DI water to rinse residues and sediments from the sample pathway.

14.1.19 Turn off the lamp.

14.1.20 Dispose of the standard and sample remaining in the tubing. Pour in the metals waste container.

15.0 CALCULATIONS

15.1 Mercury = instrument reading x dilution factor

15.2 LCS % Recovery = (Determined concentration/True value) *100

15.3 RPD = $\frac{[D1 - D2]}{\frac{(D1+D2)}{2}} \times 100$

Where D1 is the original sample result and D2 is the duplicate sample result.

15.4 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMITS

16.1 Waters: 0.0002 mg/L

16.2 Solids: 0.01 mg/kg

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in EPA methods 245.2, 7470A and 7471B.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 30 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 6%. This shall be evaluated quarterly.

17.6 Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The analyst shall evaluate quality control criteria during or immediately following analysis. Contact the supervisor immediately if criteria are not met and there is insufficient sample to repeat the test. If quality control criteria are not met and the test cannot be repeated due to insufficient sample volume or hold time, the data may be reported with a case narrative. Alternatively, contact the client to receive additional instructions such as analysis of a retain sample or authorization for repeating analysis outside of holding time.

19.2 The Method Blank concentration shall not be exceed ½ the LOQ or 1/10 the amount measured in any sample (whichever is greater), or otherwise affect sample results. High failures may also be reported with a narrative if the associated samples are non-detect. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be reanalyzed or reprepped and reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately.

19.3 The LCS control limits of 80-120% are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria for any analyte, the source of inaccuracy should be investigated and the associated batch should be reanalyzed or reprepped and reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately. If mercury is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.4 The MS is used to determine sample matrix effect. The control limits used to evaluate the MS are the same as the applicable LCS limits. If the recovery is outside of control limits and a lab error is suspected, repeat the MS determination. If the LCS is within control limits and matrix interference is indicated, report the results with a case narrative.

19.5 The Duplicate RPD should be ≤20. If the RPD is greater than the 20, investigate the source of the precision error. A source of precision error in the DUP or MS/MSD may be the homogenous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.6 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.7 Samples collected in South Carolina shall not be reported with any QC failures. The method blank must be less than the reporting limit and the LCS must be within control limits for all analytes.

19.8 Project specific criteria may apply. See SOP GEN-019.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 7470A
22.2 EPA 7471B
22.3 EPA 245.2

23.0 METHOD MODIFICATIONS

23.1 The concentration of hydroxylamine sulfate specified for method 245.2 is 12%. This procedure uses a concentration of 3% to match methods 7470A and 7471B.
23.2 Adjusted the amount of hydroxylamine sulfate added to water samples.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
10/26/12	18	Previous version	
12/05/13	19	Updated format and added sections required for TNI-compliant SOPs. Removed requirement for serial dilution. Updated calibration criteria to match method criteria. Updated initial sample sequence to reflect CCV/CCB every 10 samples and at the end of analysis. Updated MB criteria. Updated LCS criteria to reflect method criteria of 80-120%. Adjusted the amount of hydroxylamine sulfate added for water and solid samples.	Shelley Bourgeois

STANDARD OPERATING PROCEDURE FOR

METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS)

EPA 200.8 / EPA 6020A

Approved by: _____ Date: 3/7/14
R. Jason Olivier, Inorganic Manager

Approved by: _____ Date: 3/7/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 3/7/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 This SOP covers the operation of the Agilent 7700 ICP-MS for the analysis of low-level concentrations of metals. Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of concentrations of a large number of elements in water samples and in waste extracts or digests. The procedure is applicable for the analysis of aqueous samples, soils, solids, and domestic or industrial wastes for total metals, after preparation by the appropriate EPA procedure. Dissolved metals, filtered and acid preserved prior to analysis may be analyzed without digestion. Elements currently analyzed using these methods are listed in Table 1.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water, Solid, and Waste

3.0 SUMMARY OF TEST METHOD

3.1 Prior to analysis, samples should be solubilized or digested using the appropriate sample preparation methods (see GCAL SOPs MET-004, MET-005, and MET-020). When analyzing groundwater or other aqueous samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

3.2 This method describes the multi-elemental determination of analytes by ICP-MS in environmental samples. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol is transported by argon gas into the plasma torch. The ions produced by high temperatures are entrained in the plasma gas and extracted through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a mass spectrometer. The ions transmitted through the mass spectrometer are quantified by an electron multiplier and the ion information is processed by the instrument's data handling system. A collision/reaction cell utilizing He and (optionally) H_2 gases is used to remove molecular interferences. Interferences not eliminated by the collision/reaction cell must be assessed and valid corrections applied or the data qualified to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC) as described in SOP GEN-007.

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and cannot be resolved or corrected for by the mass spectrometer in use. These interferences are addressed by the instrument operating conditions, correction equations, and careful selection and monitoring of mass-to-charge ratios. In general, all direct isobaric interferences are avoided by choosing an interference-free isotope. In some cases this may result in a loss in sensitivity.

6.2 Physical Interferences are generally considered to be effects associated with the sample nebulization and transport processes. Properties such as change in viscosity and surface tension can cause significant inaccuracies especially in samples that may contain high dissolved solids or acid concentrations. If these types of interferences are present, they must be reduced by diluting the sample. Dissolved solids greater than 0.2% w/v cause interferences and clogging of the pump. Internal standards are used to identify this type of interference.

6.3 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom with the same nominal charge-to-mass ratio of the ions of interest. Proper tuning of the plasma and torch conditions reduce the formation of oxides and thus reduces this interference. In addition, the use of a collision cell reduces or eliminates this interference in most matrices.

6.4 Signals from relatively abundant isotopes can coalesce at the wings of relatively less abundant isotopes leading to loss of resolution and poor quantitation.

6.5 Memory interferences of isotopes from previous sample runs. The rinse period between samples must be long enough to eliminate significant memory interference.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when

working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Agilent 7700x ICP-MS

8.2 The Agilent 7700x ICP-MS utilizes Agilent software. User guides for the instrument, autosampler, and data acquisition software are listed below. For troubleshooting help, please contact GCAL's IT Director or Agilent.

8.2.1 Agilent 7700 Series ICP-MS, Rev. A, June 2009

8.2.2 Agilent 7700/7500 Series ICP-MS ASX-500 Series Autosampler, Rev. A, June 2009

8.2.3 Agilent 7700 Series ICP-MS Mass Hunter Workstation user guide, Rev. A, June 2009

8.3 Edwards Vacuum Pump Model E2M18

8.4 Agilent ASX-500 Autosampler

8.5 Peristaltic pump and tubing

8.6 Argon and Helium supply with pressure controls

8.7 Water purification system, Millipore Synergy Ultrapure or equivalent system to produce Type 1 water (18.2 MΩ·cm at 25 °C ultrapure water)

8.8 Class "A" volumetric flasks, various sizes

8.9 Graduated 15 mL centrifuge tubes

8.10 50 mL graduated polypropylene conical tubes

8.11 10 L Nalgene bottles

8.12 Eppendorf pipettes, various sizes, calibrated daily when in use

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Type 1 Deionized (DI) water

9.1.2 Concentrated HNO_3 – Ultra Trace metals grade

9.1.3 Concentrated HCl – Ultra Trace metals grade

9.1.4 ICP-MS rinse solution (5%HNO_3/2%HCl) – Add approximately 500 mL of type 1 DI water to a 1L volumetric flask and then add 50 mL of

concentrated HNO_3 ultra trace metals grade and 20 mL of concentrated HCl ultra trace metals grade. Bring to a final volume of 1L using type 1 DI water.

9.1.5 ICP-MS matrix solution (2%HNO_3/0.5% HCl) – Add approximately 500 mL of type 1 DI water to a 1L volumetric flask and then add 20 mL of concentrated HNO_3 ultra trace metals grade and 5 mL of concentrated HCl ultra trace metals grade. Bring to a final volume of 1L using type 1 DI water.

9.1.6 2%HNO_3 – Add approximately 500 mL of type 1 DI water to a 1L volumetric flask and then add 20 mL of concentrated HNO_3 ultra trace metals grade. Bring to a final volume of 1L using type 1 DI water.

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 ICP-MS Stock Tuning solution (10 ppm Ce, Co, Li, Tl, and Y, Part #5188-6564) – Commercially prepared and purchased from Agilent Technologies.

9.2.2 Tune check solution (10 ppb) – Prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50ml polypropylene conical tube and then using an Eppendorf pipette add 50uL of ICP-MS Stock Tuning solution. Bring to a final volume of 50mL using ICP-MS matrix solution.

9.2.3 7500 series P/A Tuning 1 and 2 (various concentrations, Part #5188-6524) – Commercially prepared and purchased from Agilent Technologies.

9.2.4 P/A tuning solution (various concentrations) – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 500 µL each of P/A Tuning 1 and 2. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.5 ICP-MS internal standard mix (100 mg/L Bi, Ge, In, Li^6, Lu, Rh, Sc, and Tb, Part # 5188-6525) – Commercially prepared and purchased from Agilent Technologies.

9.2.6 Internal Standard solution (2 ppm) – Prepared by adding approximately 100 mL of ICP-MS matrix solution to a 200 mL volumetric flask. Then add 4 mL of ICP-MS internal standard mix and bring to a final volume of 200 mL using ICP-MS matrix solution.

9.2.7 GCAL 8 Stock Calibration Standard Custom Mix, commercially prepared.

9.2.8 Stock Calibration Standards, individual metals, 1000 mg/L each, commercially prepared.

9.2.9 Working Standards, individual metals

9.2.9.1 Silver (Ag), 10 mg/L – Add 500 µL of 1000 mg/L Ag stock standard to a 50 mL polypropylene conical tube and bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.9.2 Antimony (Sb), 10 mg/L – Add 500 µL of 1000 mg/L Sb stock standard to a 50 mL polypropylene conical tube and bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.9.3 Selenium (Se), 1.0 mg/L – Add 50 µL of 1000 mg/L Se stock standard to a 50 mL polypropylene conical tube and bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.10 Interference Check Standards Stock Solutions – prepared and purchased commercially from High Purity Standards at various concentrations, catalog #ICP-MS-ICS-2 (A&B); alternatively, may be prepared from certified single element solutions. Working standard expires according to the manufacturer's expiration date or 7 days after preparation whichever is sooner or if comparisons with quality control check standards indicate a problem.

9.2.10.1 ICSA – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 50 µL of ICSA spike. Bring to a final volume of 50mL using ICP-MS matrix solution.

9.2.10.2 ICS Sn/Sr – prepared by adding approximately 40 mL ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 500 µL each of a 1000 ppm certified solution of Sn and a 1000 ppm certified solution of Sr. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.10.3 ICSAB – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 50 µL of ICSA spike, ICSB spike, and ICS Sn/Sr. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.11 Calibration Standards – Calibration standards are prepared from custom multi-element solutions or certified single element solutions. Each day of analysis, the standards are diluted to working levels using ICP-MS matrix solution. At a minimum a 3-point curve must be used.

9.2.11.1 Calibration L3 (Calibration Standard) – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 500 µL of GCAL 8, 500 µL of 10 mg/L Ag working standard, 500 µL of 10 mg/L Sb working standard, 2.5 mL of 1 mg/L Se working standard, and 20 µL of 1000 mg/L Mn stock standard. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.11.2 Calibration L2 (CCV) – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 5 mL Calibration L3 and 250 µL of 1 mg/L Se working standard. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.11.3 Calibration L1 – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 5 mL Calibration L2. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.11.4 Calibration 200.8 L1 (Al) – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 5 mL Calibration L1. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.11.5 Calibration 200.8 L1 (Be, Ag, Tl) – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 2.5 mL Calibration L2. Bring to a final volume of 50 mL using ICP-MS matrix solution.

9.2.12 Initial Calibration Verification (ICV) standards – Stock ICV standards are purchased as custom multi-element solutions or certified single element solutions. The ICV must be prepared in the same acid matrix as the calibration standards (ICP-MS matrix solution). This solution must be prepared from an independent or second source standard and be near the midpoint of the linear range.

9.2.12.1 ICV Custom Mix, commercially prepared

9.2.12.2 ICV Stock Silver Standard, 1000 mg/L, commercially prepared.

9.2.12.3 ICV Working Standard, Silver (Ag), 10 mg/L – Add 500 µL of 1000 mg/L Ag ICV stock standard to a 50 mL polypropylene conical tube and bring to a final volume of 50 mL using 2% HNO_3.

9.2.12.4 ICV – prepared by adding approximately 40 mL of ICP-MS matrix solution to a 50 mL polypropylene conical tube and then using an Eppendorf pipette add 250 µL of ICV Custom Mix and 250 µL of 10 mg/L Ag ICV working standard. Bring to a final volume of 50 mL using ICP-MS matrix solution.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.

10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.

10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.

10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation

11.1.1 Total metals, aqueous samples – HNO_3 to pH <2.

11.1.2 Dissolved metals – field filtered through a 0.45 µm filter; HNO_3 to pH <2

11.1.3 Total metals, solid and waste samples – none

11.2 Maximum holding time – 6 months from collection to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be prepared per batch, a maximum of ten samples for method 200.8 and a maximum of twenty samples for method 6020A. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be prepared per batch, a maximum of ten samples for method 200.8 and a maximum of twenty samples for method 6020A. The LCS is a known amount of standard added to type 1 DI water and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control. A LCSD is prepared if insufficient sample volume is provided to prepare a MS/MSD.

12.3 One matrix spike (MS) shall be analyzed per batch, a maximum of ten samples for method 200.8 and a maximum of twenty samples for method 6020A.

12.4 One laboratory duplicate (DUP) or matrix spike duplicate (MSD) shall be analyzed per batch, a maximum of ten samples for method 200.8 and a maximum of twenty samples for method 6020A.

12.5 Post Digestion Spike – A post digestion spike is performed if MS/MSD recoveries for target analytes are outside control limits. Post digestion spikes are not required by EPA method 200.8. All MS/MSD failures will be reported with a narrative.

12.6 Dilution test (serial dilution) – If the Post Digestion Spike fails, perform a 5x dilution on the QC sample. The concentration of the analytes in the sample to be diluted should be greater than 50 times the MDL. Dilution tests are not required by EPA method 200.8. All MS/MSD failures will be reported with a narrative.

12.7 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Tuning criteria – Prior to performing the initial calibration the instrument must pass the mass calibration, resolution, and stability or tune check. The mass calibration cannot differ by more than 0.1 amu from the true value. The resolution must also be verified to be less than 0.75 amu full width at 5% peak height. For stability, the % RSD must be ≤5% for at least 5 replicate analyses. If these criteria

are not achieved, perform instrument maintenance and/or tuning. Calibration and sample analysis cannot begin until the aforementioned tuning criteria are met.

13.2 Initial Calibration (ICAL) – The initial calibration consists of a calibration blank and a minimum of three contiguous non-zero points. The internal standard response from the calibration blank is used to calculate drift for all samples in the analytical batch. Additional points may be analyzed to better define the linear range and the lower limit of quantitation. The low or high points may be removed from a multi-point curve as long as a minimum of three points remain. Each analyte must exhibit a correlation coefficient (r) of 0.998 to be considered acceptable. A successful initial calibration curve must be analyzed before any result can be quantitated. A new curve must be performed at a minimum daily.

13.3 Initial Calibration Verification (ICV) – Immediately following the initial calibration or prior to sample analysis the initial calibration is verified with a mid-level ICV standard using the acceptance criteria of 90-110% for all target analytes. The ICV standard must be prepared from a second source standard at or near the mid-range of the calibration curve. If the criteria referenced above are not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation and reanalysis of the standards involved.

13.4 Initial Calibration Blank (ICB) – The ICB is used to verify all analyte concentrations are below the client defined reporting limit. The concentration of all analytes must be ≤ the absolute value of the LOQ. For DOD projects, concentrations must be < the LOD. The ICB is prepared from ICP-MS matrix solution.

13.5 Interference Check Solution A (ICSA) – The ICSA is used to monitor correction factors for common interferents. The concentration for all non-spiked analytes must be ≤ the absolute value of the LOQ. For DOD projects, concentrations must be < the absolute value of the LOD. The ICSA is analyzed before sample analysis and once every 12 hours.

13.6 Interference Check Solution B (ICSAB) – All spiked analytes in the ICSAB solution must have recoveries of 80-120%. The ICSAB is analyzed before sample analysis and once every 12 hours.

13.7 Continuing Calibration Blank (CCB) – The CCB is prepared using the ICP-MS matrix solution and analyzed after every 10 samples (or more frequently) and at the end of the analytical analysis. The concentration of all analytes must be ≤ the absolute value of the LOQ. For DOD projects, concentrations must be < the LOD. The CCB is prepared from ICP-MS matrix solution. Data is reportable with a narrative if an analyte is detected above the reporting limit in the CCB provided the analyte is not detected during the associated sample analysis or if the concentrations of the analyte in the associated samples are greater than 10x the concentration of the analyte in the CCB.

13.8 Low Level Continuing Calibration Verification (LLCCV) – The calibration standard at the LOQ level is also analyzed as the LLCCV. The LLCCV is

analyzed at the beginning of analysis and after every 10 samples (or more frequently) and at the end of analysis. The recovery for all target analytes in the initial LLCCV must be 80-120%. The recovery for all target analytes in each subsequent LLCCV must be 70-130%.

13.9 Continuing Calibration Verification (CCV) – The mid-level calibration standard is also analyzed as the CCV. The CCV is analyzed after every 10 samples (or more frequently) and at the end of the analytical analysis. The recovery all target analytes in the CCV is 90-110%.

13.10 Samples reported by SW-846 6020A shall be diluted for any compound greater than the high level calibration standard. Samples with analyte concentrations greater than 90% of the high level ICAL will be diluted and reanalyzed for samples reported by EPA method 200.8. Dilute so that the expected concentration is approximately in the mid-level of the curve. Report multiple dilutions if requested by the client.

13.11 Internal standards (IS) – Internal standards are monitored for every analysis and are quantitated against the calibration blank (see Sec 13.2). The internal standard for each QC sample and field sample must meet the recovery specified by the method of analysis: 70-130% for method 6020A, and 60-125% for method 200.8, of the original response in the calibration blank. For DOD samples, the IS for each sample must be 30-120%. Recoveries outside of the acceptable range may be due to instrument drift, sample matrix, or instrumentation malfunction. Instrument drift may be evaluated by reviewing internal standard recoveries in a clean matrix (CCB). Extremely high recoveries for the internal standards can result from the presence of the internal standard element in the original sample and may be accounted for by evaluating the sample using an alternate acceptable internal standard. Clogged sample transfer lines or other types of instrument malfunctions or exposure can cause unacceptable internal standard recoveries. A sample must be reanalyzed if an internal standard recovery is outside of the acceptance range. If the internal standard failure is determined to be due to sample matrix, the sample should be reanalyzed at a dilution five times greater than the original analysis in which the internal standard recovery was unacceptable.

13.12 Determine DLs using the requirements of SOP QA-009.

14.0 PROCEDURE

14.1 Allow the instrument to become thermally stable for at least 30 minutes before tuning.

14.2 Tune the instrument with the tune check solution (sec 9.2.5). See section 13.1 for tune acceptability.

14.3 The ICP-MS is calibrated each day of operation using a blank and at least 3 standards. See section 13.2 for ICAL criteria.

14.4 An ICV standard is analyzed immediately after the initial calibration or prior to sample analysis. This ICV standard is prepared from a second source. See section 13.3 for acceptance criteria.

14.5 An ICB is analyzed after the ICV to check for memory effects. See section 13.4 for acceptance criteria.

14.6 Interference Checks ICSA and ICSAB are analyzed to check inter-element corrections. See section 13.5 and 13.6 for acceptance criteria.

14.7 Sample Analysis

14.7.1 Report the average of all field and QC samples analyzed.

14.7.2 Flush the system with a rinse solution (3 rinse solutions used) between samples and standards during the analytical run.

14.7.3 If a target analyte is detected above the linear calibration range, the sample must be diluted sufficiently to bring the target analyte concentration to within the calibration range.

14.7.4 The analytical run sequence should be performed as follows:

- Instrument initialization
- Warm-up
- Tune Instrument
- Tune check (mass calibration, resolution and stability check)
- Calibration blank
- Initial calibration
- ICV
- ICB
- LLCCV (at LOQ)
- ICSA
- ICSAB
- Samples (includes investigative samples and batch QC such as MB, LCS, MS, MSD, etc.)
- CCV Block (CCB, LLCCV, CCV see sections 13.7-13.9) every 10 samples
- ICSA and ICSAB at a minimum every 12 hrs.
- Closing CCV Block
- Termination Block

15.0 CALCULATIONS

15.1 Water samples (µg/L) = Instrument reading x DF

15.2 Soil samples (µg/Kg) = (Instrument reading x DF x FV) / Sample Weight (g)

Where: DF = dilution factor

FV = final extract volume (mL)

15.3 % Recovery (MS/MSD) = $\frac{\text{Spike Concentration-Sample Concentration x 100}}{\text{Spike Added}}$

15.4 % Recovery (LCS/LCSD) = $\frac{\text{Spike Concentration x 100}}{\text{Spike Added}}$

15.5 RPD = $\frac{|\text{Concentration 2-Concentration 1}| \times 100}{(\text{Concentration 2-Concentration 1})/2}$

15.6 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMITS

16.1 LOQ for water and soil samples – see Table 1

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in EPA methods 200.8 and 6020A.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 30 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 6%. This shall be evaluated quarterly.

17.6 Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The analyst shall evaluate quality control criteria during or immediately following analysis. Contact the supervisor immediately if criteria are not met and there is insufficient sample to repeat the test. If quality control criteria are not met and the test cannot be repeated due to insufficient sample volume or hold time, the data may be reported with a case narrative. Alternatively, contact the client to receive additional instructions such as analysis of a retain sample or authorization for repeating analysis outside of holding time.

19.2 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the on-column amount measured in any sample (whichever is greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code “RP” for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-

prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately.

19.3 The LCS control limits of 85-115% for 200.8 and 80-120% for 6020A are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria for any analyte, the source of inaccuracy should be investigated and samples should be re-extracted and/or reanalyzed. Enter condition code "RP" for each sample affected, a new prep and analytical schedule will populate in the sample when saved. If additional sample is not available or the samples were re-prepared outside of holding time, report with a case narrative and notify the GCAL project manager immediately. If a target compound is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.4 The MS is evaluated against the LCS control limits and is used to determine sample matrix effect. If the MS recovery is outside of control limits and the LCS is within control limits, matrix interference is indicated. Report the results with a case narrative and perform a post digestion spike.

19.5 The Duplicate RPD should be ≤20. If the RPD is outside the control limits, investigate the source of the precision error. A source of precision error in the DUP or MS/MSD may be the heterogenous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.6 A post digestion spike is performed if MS/MSD recoveries for target analytes are outside control limits. A spike is added to a portion of a digested sample or its dilution and should be recovered at 80-120% of the known value. If a recovery is outside control limits, perform the dilution test.

19.7 The serial dilution neat sample concentration and 5 times the diluted sample concentration should agree within 10%. If not, a chemical or physical interference should be suspected.

19.8 Review data to verify that an analytical error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.9 Native sample concentrations may be high in comparison to the spiking concentration and therefore an accurate recovery cannot be calculated. Document this in the case narrative.

19.10 Spikes may be diluted out in the analysis process if a 10X or higher dilution is performed. Document this in the case narrative.

19.11 Project specific criteria may apply. See SOP GEN-019.

19.12 For data review procedures and checklists, see SOP QA-002.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 EPA 200.8, Revision 5.4

22.2 EPA Method 6020A, Revision 1, February 2007

22.3 Agilent application note *Unmatched Removal of Spectral Interferences in ICP-MS Using the Agilent Octopole Reaction System with Helium Collision Mode*

23.0 METHOD MODIFICATIONS

23.1 None

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history

24.2 Table 1 – Analytes and Reporting Limits

24.3 Table 2 – Calibration Levels

24.4 Attachment 1 – ICP-MS Cover Page

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
08/24/12	03	Previous version	
12/06/13	04	Updated format and added sections required for TNI-compliant SOPs. Clarified requirement for initial LL CCV. Updated detection limits for Na. Deleted Zr from list of analytes. Added concentrations of calibration standards.	Shelley Bourgeois
03/07/14	05	Updated control limits for internal standards (13.11). Updated calibration tables. Corrected typos. Updated standards and reagents to include all individual calibration standards. Corrected Se concentration in calibration Table 2.	Shelley Bourgeois Abigail Guerin

Table 1 – Analytes and Reporting Limits

Analyte	6020A Waters, µg/L	6020A Soils, µg/kg	200.8 Waters, µg/L
Aluminum	20	800	2.5
Antimony	2	80	2
Arsenic	1	40	1
Barium	1	40	1
Beryllium	1	40	0.5
Cadmium	1	40	1
Calcium	500	20000	500
Chromium	1	40	1
Chromium (III)	1	40	1
Cobalt	1	40	1
Copper	1	40	1
Iron	100	4000	100
Lead	1	40	1
Magnesium	100	4000	100
Manganese	5	200	5
Molybdenum	1	40	1
Nickel	2	80	2
Potassium	100	4000	100
Selenium	1	40	1
Silver	1	40	0.5
Sodium	100	4000	100
Strontium	1	40	1
Thallium	1	40	0.5
Tin	1	N/A	1
Titanium	1	N/A	1
Vanadium	1	40	1
Zinc	20	800	20

Table 2 – Calibration Levels

Analyte	200.8 L1	Calibration L1	Calibration L2	Calibration L3
Aluminum	2.0	20	200	2000
Antimony		2	20	200
Arsenic		1	10	100
Barium		1	10	100
Beryllium	0.5	1	10	100
Cadmium		1	10	100
Calcium		500	5000	50000
Chromium		1	10	100
Chromium (III)		1	10	100

Cobalt		1	10	100
Copper		1	10	100
Iron		100	1000	10000
Lead		1	10	100
Magnesium		100	1000	10000
Manganese		5	50	500
Molybdenum		1	10	100
Nickel		2	20	200
Potassium		100	1000	10000
Selenium		1	10	50
Silver	0.5	1	10	100
Sodium		100	1000	10000
Strontium		1	10	100
Thallium	0.5	1	10	100
Tin		1	10	100
Titanium		1	10	100
Vanadium		1	10	100
Zinc		20	200	2000

ICP-MS

BY METHODS EPA 200.8/6020A

Date: _____
Analyst: _____

Instrument ID: _____
HBN: _____
Batch: _____

Cal Standard _____ Exp. Date _____

ICV Standard _____ Exp. Date _____

ICSA Standard _____ Exp. Date _____

ICSAB Standard _____ Exp. Date _____

Internal Standard _____ Exp. Date _____

Tune _____ Exp. Date _____

P/A _____ Exp. Date _____

ICPMS Acid Matrix (Calibration Blank) (2% HNO3/0.5% HCl) _____ Exp. Date _____

ICPMS Acid Matrix (5% HNO3/2% HCl) _____ Exp. Date _____

QC Limits:

Calibration Correlation Coefficient R ≥ 0.998

	GCAL Limits
ICV	90-110%
LLCCV (initial)	80-120%
ICSA/ICSAB	80-120%
LLCCV (continuing)	70-130%
CCV	90-110%

Reviewer/Date:_____

STANDARD OPERATING PROCEDURE FOR

HEXAVALENT CHROMIUM

SM 3500-Cr B-2011
EPA 7196A

Approved by: _____ Date: 5/16/14
R. Jason Olivier, Inorganic Manager

Approved by: _____ Date: 5/16/14
Scott Bailey, Laboratory Manager

Approved by: _____ Date: 5/16/14
Shelley Bourgeois, Quality Assurance Manager

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1.0 SCOPE AND APPLICATION

1.1 Hexavalent chromium in water samples or soil/solid digestates is determined through colorimetric development by reaction with diphenylcarbazide in acid solution with analysis by colorimetric spectrophotometer.

2.0 APPLICABLE MATRIX OR MATRICES

2.1 Water, Solid

3.0 SUMMARY OF TEST METHOD

3.1 Solid samples are digested using an alkaline digestion to solubilize most hexavalent chromium compounds without altering the chromium oxidation states.

3.2 Aqueous samples and digestates of solid samples are analyzed using colorimetric analysis at 540 nm following reaction with diphenylcarbazide in acid solution to form a red-violet colored complex.

4.0 DEFINITIONS

4.1 Refer to SOP GEN-016

5.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

5.1 The analyst performing this test procedure must have completed the required safety and technical orientation, ethics training, on-the-job training, and an initial demonstration of capability (IDOC).

5.2 The analyst must have completed a demonstration of capabilities (DOC) as described in SOP GEN-007. The demonstration of capabilities must be completed annually.

6.0 INTERFERENCES

6.1 Reduction and/or oxidation reactions constitute the primary interference associated with the digestion of solid samples.

6.2 The method for water samples is relatively free of interferences.

7.0 SAFETY

7.1 Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO the use of any chemical. Employees must be familiar with the applicable material safety data sheets (MSDS). Chemical labels on the containers or packages contain important information on the safe handling, fire hazard and health hazards associated with the chemical.

7.2 Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. Exposure to these materials must be reduced to the lowest level possible. Therefore, work should be performed in well ventilated areas in a fume hood.

7.3 The appropriate personal protective equipment (PPE) must be worn when working in the laboratory. For this procedure the required PPE is a lab coat, eye covering (safety glasses or goggles) and gloves; the PPE shall be worn when handling samples and/or chemicals.

7.4 Contact lenses are not to be worn in the laboratory.

7.5 Food or drink may not be present when actively processing or handling samples.

8.0 EQUIPMENT AND SUPPLIES

8.1 Digestion (solid samples)

- 8.1.1 125 mL beakers or flasks
- 8.1.2 25 and 125 mL graduated cylinders
- 8.1.3 Vacuum flasks
- 8.1.4 Filtering funnels
- 8.1.5 Filter membranes – 0.45µm
- 8.1.6 Stirrer/Hotplate – capable of heating to 90-95ºC with continuous auto stirring capability
- 8.1.7 Teflon stir bars
- 8.1.8 Calibrated pH meter & buffers
- 8.1.9 Calibrated ORP meter
- 8.1.10 Electronic balance – capable of weighing to 0.01 g, calibrated daily when in use
- 8.1.11 Thermometer – NIST traceable
- 8.1.12 Eppendorf pipette, calibrated daily when in use
- 8.1.13 Plastic cups
- 8.1.14 Specimen cups
- 8.1.15 Spatula
- 8.1.16 Wooden tongue depressors
- 8.1.17 Weigh paper
- 8.1.18 Parafilm
- 8.1.19 Blank sand or equivalent

8.2 Analysis (aqueous samples and solid sample extracts)

- 8.2.1 HACH DR2800 Spectrophotometer set at 540nm
- 8.2.2 The HACH instrument utilizes HACH DATA TRANS Version 1.0.2.0 to transfer data from the HACH DR2800 instrument to an Excel spreadsheet and ultimately to the Horizon LIMS. If any problems arise with the data transfer, contact GCAL's IT Director for troubleshooting.
- 8.2.3 25 mL sample cell for HACH
- 8.2.4 25 mL graduated cylinder
- 8.2.5 Eppendorf pipette, calibrated daily when in use
- 8.2.6 100 mL volumetric flask
- 8.2.7 Plastic Cups
- 8.2.8 Timer

9.0 REAGENTS AND STANDARDS

9.1 Reagents – all reagents are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. When the manufacturer fails to provide an expiration date, the expiration date will be 12 months from the date opened or sooner if the reagents show signs of deterioration such as change in color, clumping, separation, etc. If tests or appearance indicates the reagent has decomposed or is contaminated, dispose of the reagent and notify the QA Department and/or supervisor. Label all reagents with reagent ID, lot number, date received, date opened and expiration date. Label all prepared solutions as required by SOP GEN-006.

9.1.1 Deionized (DI) water

9.1.2 Nitric acid, concentrated

9.1.3 Nitric acid solution (1:1)

9.1.4 Sodium carbonate, anhydrous

9.1.5 Sodium hydroxide solution (50%)

9.1.6 Magnesium chloride (anhydrous), analytical reagent grade – 392.18 mg $MgCl_2$ is equivalent to 100 mg Mg^{2+}.

9.1.7 Potassium phosphate, dibasic

9.1.8 Potassium phosphate, monobasic

9.1.9 Phosphate Buffer (0.5M K_2HPO_4/0.5M KH_2PO_4 buffer at pH 7 – Dissolve 87.09 g K_2HPO_4 and 68.04 g KH_2PO_4 into a 1L volumetric flask filled approximately half way with DI water, and dilute to volume with DI water.

9.1.10 Digestion solution – Dissolve 40.0 ± 0.05 g 50% NaOH and 30.0 ± 0.05 g Na_2CO_3 into a 1L volumetric flask filled approximately half way with DI water, and dilute to volume with DI water. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater; if not, discard. This solution expires 1 month from the date made.

9.1.11 HACH ChromaVer ® 3 Chromium Reagent Powder Pillows. The reagent contains an acidic buffer combined with 1,5-Diphenylcarbohydrazide. The contents of the reagent are:

9.1.11.1 Potassium pyrosulfate

9.1.11.2 Magnesium Sulfate

9.1.11.3 1,5-Diphenylcarbohydrazide

9.2 Standards – all standards used must be pure material or from prepared certified solutions. A copy of the accompanying certificate of analysis (CoA) must be kept on file. Stock standards are to be stored according to manufacturer's instruction and will expire on the date specified by the manufacturer. Label all working standards with date prepared, concentration, standard ID number, expiration date and the initials of individual preparing the standard. Record all standard preparations in the Standard Preparation Logbook. Label prepared standards as described in SOP GEN-006.

9.2.1 Stock Standard (commercially prepared, chromium, 50 mg/L)

9.2.2 Insoluble Matrix Spike (commercially prepared, lead chromate)
9.2.3 Stock ICV Standard (commercially prepared, chromium, 1000 mg/L)
9.2.4 Working ICV Standard (10 mg/L) – Add 0.5 mL of stock ICV standard to a 50 mL volumetric flask and dilute to volume with DI water. Prepare fresh each use.
9.2.5 ICV (0.5 mg/L) – Add 1.25 mL of working ICV standard to a 25 mL volumetric flask and dilute to volume with DI water. Prepare fresh each use.
9.2.6 Calibration Standards – The calibration standards are prepared by diluting the 50 mg/L stock standard into 5 working levels. The standards are diluted with DI water.
9.2.6.1 Calibration Standard 1 (0.01 mg/L) – Add 20 µL of stock standard to a 100 mL volumetric flask and dilute to volume with DI water.
9.2.6.2 Calibration Standard 2 (0.05 mg/L) – Add 100 µL of stock standard to a 100 mL volumetric flask and dilute to volume with DI water.
9.2.6.3 Calibration Standard 3 (0.10 mg/L) – Add 200 µL of stock standard to a 100 mL volumetric flask and dilute to volume with DI water.
9.2.6.4 Calibration Standard 4 (0.50 mg/L) – Add 1000 µL of stock standard to a 100 mL volumetric flask and dilute to volume with DI water.
9.2.6.5 Calibration Standard 5 (1.0 mg/L) – Add 2000 µL of stock standard to a 100 mL volumetric flask and dilute to volume with DI water.

10.0 SAMPLE COLLECTION, SHIPMENT, AND STORAGE

10.1 Samples are received at the laboratory by common carrier or hand delivered. Sample receipt policies are described in SOP SAD-001.
10.2 A chain-of-custody must accompany all samples and must document the date and the time of sample collection and the preservation method used. Internal chain of custody procedures are described in SOP SAD-002.
10.3 Samples are collected in plastic or glass containers and stored at the laboratory using conditions required by 40CFR 136 or reference method.
10.4 Samples are stored at the laboratory in a secure location until disposal.

11.0 PRESERVATION AND HOLD TIME

11.1 Required preservation – Cool to 0-6° C, but not frozen
11.2 Maximum holding time
11.2.1 Waters – 24 hours from collection to analysis
11.2.2 Soils – 30 days from collection to digestion; 7 days from digestion to analysis

12.0 QUALITY CONTROL

12.1 One method blank (MB) shall be analyzed per batch, a maximum of twenty samples. The blank is prepared in the exact manner of the samples, and is analyzed to demonstrate that the extraction and/or analytical procedure did not introduce contamination.

12.2 One laboratory control sample (LCS) shall be analyzed per batch, a maximum of twenty samples. The LCS is a known amount of standard added to DI water and prepared in the exact manner of the samples, and is analyzed to demonstrate that preparation and analytical systems are in control.

12.3 One matrix spike (MS) shall be analyzed per batch, a maximum of twenty samples. The MS is a known amount of standard added to a sample at the same concentration as the LCS.

12.4 One insoluble matrix spike (MS) shall be analyzed with each batch of soil samples.

12.5 One laboratory duplicate (DUP) and/or matrix spike duplicate (MSD) shall be analyzed per batch, a maximum of twenty samples.

12.6 Project specific criteria may apply. See SOP GEN-019.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Initial Calibration (ICAL) – an ICAL is performed using a blank and 5 standards. An initial calibration curve must be analyzed and evaluated before any result can be quantitated. A new curve must be performed when the ICV or CCV acceptance criteria is not met, the standard expires, when major maintenance is performed, or at least annually.

13.1.1 Allow standards to come to room temperature, and then prepare calibration standards as indicated in Procedure 9.2.5.

13.1.2 Measure 25 mL of DI water for the calibration blank and 25 mL of each standard. Pour into a plastic cup and add the contents of one ChromaVer® 3 powder pillow to each cup and swirl for 20 seconds. Allow color to develop for 5 minutes.

13.1.3 On the HACH instrument:

13.1.3.1 Select “User Program”

13.1.3.2 Highlight “CROM6”

13.1.3.3 Select “Program options”

13.1.3.4 Select “Edit”

13.1.3.5 Select “Calibration c=a+AB” and press “Edit”

13.1.3.6 Select “Read Standards” and press “OK”

13.1.3.7 Read the blank and each of the standards by transferring each to a sample cell on the instrument and selecting “Read”

13.1.3.8 Press “Graph” and check the slope

13.1.3.9 Select “Done”, “Store”, and “Cancel”. The instrument will display the question “Store Program?” Select “Yes”.

13.1.3.10 Print the graph and the calibration table.

13.1.3.11 Enter the calibration data into the HACH Initial Calibration Spreadsheet (Attachment 2).

13.1.4 The correlation coefficient must be 0.995 or greater.

13.2 Immediately following each calibration, a blank must be analyzed and the result must be less than ± one half the Limit of Quantitation (LOQ).

13.3 Following each ICAL, the low-level calibration standard (0.01 mg/L) must be analyzed and the recovery must be ±50% (50-150% of true value). For projects originating in South Carolina, the low-level calibration check standard must be analyzed with each batch.

13.4 Independent Calibration Verification (ICV) – immediately following the ICAL analyze a standard from a solution that is independent from the ICAL standard. It is preferred that the ICV be from a different manufacturer. If this is not available then a different lot from the same manufacturer may be used.

13.5 The ICV is prepared at a concentration of 0.5 mg/L, and the recovery must be ±10% (90-110% of true value).

13.6 If the criterion referenced above is not achieved, corrective action must be taken. This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved.

13.7 Continuing Calibration Verification (CCV) – each day of analysis that an ICAL is not performed a CCV must be analyzed before sample analysis. The CCV is also analyzed every 10 samples and at the end of the analytical batch.

13.8 The CCV is prepared at a concentration of 0.5 mg/L, and the recovery must be ±10% (90-110% of true value).

13.9 If the calibration verification standard fails to meet the criteria, repeat the analysis of the standard. If the standard fails again, take appropriate corrective action (inspection of HACH, re-prep standard, etc.). If the criteria cannot be met, a new calibration curve should be prepared.

13.10 If the calibration verification standard analyzed after a group of samples has a high recovery and the analyte was not detected in any of the previous samples bracketed by that CCV, then the samples do not need to be re-analyzed.

14.0 PROCEDURE

14.1 Solid Sample Preparation

14.1.1 Turn on the hotplate to the appropriate setting to obtain a temperature of 90-95ºC. Monitor a temperature blank (beaker with at least 50 mL of digestion solution) with a NIST traceable thermometer.

14.1.2 Label a clean beaker or flask with the sample ID for all samples in the batch.

14.1.3 Weigh 2.50 ± 0.2 g of blank sand into the flasks labeled MB and LCS. Add 625 µL of the 100 mg/L working standard to the LCS. An LCSD may be performed if requested by the client or required in the project plan.

14.1.4 Weigh 2.50 ± 0.2 g of well-mixed sample the flasks labeled for each sample. Record the weight on the prep log. Weigh two additional aliquots for the sample chosen as the batch matrix spike, one of which will be the soluble MS, and the other the insoluble MS. For the soluble MS, add 625 µL of the 100 mg/L working standard. For the insoluble MS, add 10-20 mg of $PbCrO_4$ standard. A matrix spike duplicate may be performed if requested by the client or required in the project plan. Weigh an additional aliquot of the sample chosen as the batch duplicate.

14.1.5 Add 25 mL of the digestion solution to each sample. Add 0.20 ± 0.01 g $MgCl_2$ and 0.25 mL of Phosphate buffer.

14.1.6 Add a stir bar to each sample. Stir the samples continuously (unheated) for at least five minutes.

14.1.7 Heat at 90-95ºC and maintain the samples at 90-95ºC for one hour with continuous stirring.

14.1.8 Cool the samples to room temperature.

14.1.9 Transfer the digestate to the filtration apparatus. Rinse the flask with DI water, and filter through a 0.45 µm membrane filter. Rinse the filter and filter funnel thoroughly with DI water. Remove the filter funnel and membrane from the filtration apparatus.

14.1.10 Place a magnetic stirring bar into the flask containing the filtrate, place the flask on a stirrer, and with constant stirring, and slowly add 1:1 Nitric acid to the flask drop wise. Adjust the pH of the solution to 7.0-8.0 and monitor the pH with a pH meter. If the pH of the digest should drop below 7.0, discard the solution and re-digest. Once the pH has been adjusted to 7.0-8.0, slowly add 1:1 Sulfuric acid to the flask drop wise to adjust the pH to 1.5-2.5. Perform this step in a hood.

14.1.11 Transfer the contents of the flask to a 125 mL graduated cylinder. Rinse the flask thoroughly and collect the rinsate in the cylinder. Dilute to a final volume of 125 mL. Cover with Parafilm and mix well. Transfer the solution back to the labeled flask and cap.

14.1.12 Print the batch prep log and record all required information. Deliver a copy of the prep log and the samples to the wet lab.

14.2 HACH Analysis of Solid Sample Extracts

14.2.1 Recall the stored program for Hexavalent chromium.

14.2.2 Analyze a CCV. If the CCV does not meet criteria, a new calibration curve must be analyzed.

14.2.3 Shake the extract to homogenize and measure 25 mL of the each digestate sample and QC sample and pour into a plastic cup labeled with the sample ID.

14.2.4 Prepare a post digestion spike by adding 250 µL of the 50 mg/L stock standard to a 25 mL aliquot of the sample selected for spiking. The spike concentration is 0.50 mg/L.

14.2.5 Add the contents of one ChromaVer® 3 powder pillow to the first sample cup and swirl for 20 seconds. Allow color to develop for 5 minutes.

14.2.6 Before reading each sample, zero the instrument using 25 mL of the sample with no reagents added. This allows the instrument to account for the turbidity and color of each sample. Discard this sample in the waste container.

14.2.7 Transfer the color developed sample into the sample cell and read on the instrument.

14.2.8 Repeat steps 14.2.5-14.2.7 for each sample.

14.2.9 Dilute samples that are more concentrated than the highest standard. If color or turbidity of the sample is interfering with analysis, document that the sample was diluted due to color.

14.3 HACH Analysis of Aqueous Samples

14.3.1 Recall the stored program for Hexavalent chromium.

14.3.2 Analyze a CCV. If the CCV does not meet criteria, a new calibration curve must be analyzed.

14.3.3 Prepare a method blank by adding 25 mL of DI water to a plastic cup.

14.3.4 Prepare an LCS (0.50 mg/L) by adding 250 µL of stock standard to a 25 mL volumetric flask and diluting to volume with DI water.

14.3.5 Allow the samples to come to room temperature. If samples have not been field filtered, filter through a 0.45 micron filter.

14.3.6 Measure 25 mL of the each water sample and QC sample and pour into a plastic cup labeled with the sample ID.

14.3.7 Measure a duplicate aliquot for the samples selected as the batch duplicate and matrix spike. Prepare the matrix spike by adding 250 µL of the 50 mg/L stock standard to a 25 mL aliquot of the sample selected for spiking. The spike concentration is 0.50 mg/L.

14.3.8 Add the contents of one ChromaVer® 3 powder pillow to the first sample cup and swirl for 20 seconds. Allow color to develop for 5 minutes.

14.3.9 Before reading each sample, zero the instrument using 25 mL of the sample with no reagents added. This allows the instrument to account for the turbidity and color of each sample. Discard this sample in the waste container.

14.3.10 Transfer the color developed sample into the sample cell and read on the instrument.

14.3.11 Repeat steps 14.3.8-14.3.10 for each sample.

14.3.12 Dilute samples that are more concentrated than the highest standard. If color or turbidity of the sample is interfering with analysis, document that the sample was diluted due to color.

15.0 CALCULATIONS

15.1 Hexavalent chromium (mg/L) = instrument reading x dilution factor

15.2 Hexavalent chromium (mg/Kg) =
(instrument reading x dilution factor) * (final prep volume (mL)/initial prep weight (g))

15.3 LCS % Recovery = (Determined concentration/True value) *100

15.4 RPD = $\frac{[D1 - D2]}{\frac{(D1+D2)}{2}} \times 100$

Where D1 is the original sample result and D2 is the duplicate sample result.

15.5 Refer to SOP GEN-021 for calibration calculations.

16.0 REPORTING LIMITS / LIMIT OF QUANTITATION (LOQ)

16.1 Water samples – 0.01 mg/L

16.2 Soil samples – 0.5 mg/Kg

17.0 METHOD PERFORMANCE

17.1 Method performance is based on the precision and accuracy determined by the laboratory using procedures in SM 3500-Cr and 7196A.

17.2 Laboratory limits shall be developed through the continued performance of this method by determining the standard deviation over at least 20 replicates. The recovery limits shall be set at ± 3*SD.

17.3 When available, a blind Proficiency Test (PT) sample shall be analyzed twice per year per matrix.

17.4 Sensitivity of the method shall be determined by determination of method detection limits and verified by the performance of LODs and LOQs. These requirements are described by SOP QA-009.

17.5 The single operator precision goal is SD ≤ 6%. This shall be evaluated quarterly.

17.6 Control charts shall be used to determine if goals are achieved and potential improvements of both method performance and analyst training.

18.0 POLLUTION PREVENTION

18.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation.

18.2 Refer to QAPP Section 10.2

19.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA

19.1 The analyst shall evaluate quality control criteria during or immediately following analysis. Contact the supervisor immediately if criteria are not met and there is insufficient sample to repeat the test. If quality control criteria are not met and the test cannot be repeated due to insufficient sample volume or hold time, the data may be reported with a case narrative. Alternatively, contact the client to receive additional instructions such as analysis of a retain sample or authorization for repeating analysis outside of holding time.

19.2 The Method Blank concentration shall not be greater than half the Limit of Quantitation (LOQ) or 1/10 the amount measured in any sample (whichever is

greater), or otherwise affect sample results. If the method blank fails to meet these criteria, the source of the contamination should be investigated and samples should be reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately.

19.3 The LCS control limits of 80-120% are used to determine batch acceptability. If the LCS fails to meet the laboratory acceptance criteria, the source of inaccuracy should be investigated and samples should be reanalyzed. If additional sample is not available, report with a case narrative and notify the GCAL project manager immediately. If hexavalent chromium is not detected in a sample and the LCS shows high bias (recovery above the upper control limit), the sample can be reported with a narrative. The analyst is still required to investigate the source of the failure.

19.4 The MS control limits of 80-120% for aqueous samples and 75-125% for solid samples are used to determine sample matrix effect. If the recovery is outside of control limits and a lab error is suspected, repeat the MS determination. If the LCS is within control limits and matrix interference is indicated, analyze a post digestion spike and report the results with a case narrative.

19.5 A post digestion spike (PDS) should be analyzed for any sample with a MS recovery outside the control limits listed in 19.4. The PDS recovery should be 85-115%. If the MS and/or PDS recoveries fall outside the acceptable control limits, additional testing may be requested on a project-specific basis. See Appendix A for additional information.

19.6 The Duplicate RPD should be ≤20 when the value of both duplicate sample aliquots is greater than or equal to five times the reporting limit. If the RPD is greater than the 20, investigate the source of the precision error. A source of precision error in the DUP or MS/MSD may be the homogenous nature of the sample. If a lab error is suspected, repeat the analysis. If a matrix issue is indicated, report the results with a case narrative.

19.7 Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before identifying a failure as matrix interference.

19.8 Project specific criteria may apply. See SOP GEN-019.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

20.1 Refer to GEN-018 for instructions on corrective actions. Corrective actions must be completed when the items below are noted:

20.1.1 Whenever a sample holding time is missed due to lab error (not applicable if samples were received with less than one day of holding time remaining or if for any reason analysis could not proceed while waiting for instructions from a client).

20.1.2 If a sample has been reported incorrectly for any reason.

21.0 WASTE MANAGEMENT

21.1 All unused samples or sample extracts are discarded according to the Hazardous Waste Disposal SOP, GEN-009.

22.0 REFERENCES

22.1 SM 3500-Cr B-2011
22.2 SM 3020-2011
22.3 SW-846 7196A
22.4 SW-846 3060A
22.5 HACH method 8023

23.0 METHOD MODIFICATIONS

23.1 HACH method 8023 is followed and is an accepted method for wastewater analysis of hexavalent chromium.
23.2 Method 3060A specifies digestion of a 2.5 gram aliquot to a final volume of 250 mL. This has been modified to a final volume of 125 mL. Reagents have been adjusted accordingly.
23.3 The corrective actions listed in method 3060A have been modified. Additional corrective actions may be performed based on contract requirements.

24.0 TABLES, DIAGRAMS, AND FLOWCHARTS

24.1 Document review and revision history
24.2 Appendix A – Corrective Actions for MS Nonconformances
24.3 Attachment 1 – HACH Standard ID Sheet
24.4 Attachment 2 – HACH Initial Calibration Spreadsheet

Note: Actions older than 5 years may be removed from this record.

Document Review and Revision History

Approval Date	Revision No.	Record of Activity	Author
04/05/11	10	Previous version	
06/27/13	11	Updated format and added sections required for TNI-compliant SOPs. Updated method reference to Standard Methods 22nd edition.	Shelley Bourgeois
07/18/13	12	The procedure has been adjusted for a final volume of 125 mL rather than 250 mL. The procedure was corrected to account for the pH adjustment done using the HACH powder pillow. The holding time for solid samples was changed from 3 days after extraction to 24 hours after extraction.	Shelley Bourgeois
08/26/13	13	Corrected step 14.1.10. The pH adjustment to 2.0 ± 0.5 specified in method 7196A is performed in the prep laboratory. No further pH adjustment for analysis is required.	Shelley Bourgeois
05/16/14	14	Updated hold time for solid sample extracts. Added attachments. Updated requirement for evaluation of ICAL intercept (13.2). Added the requirement for the LOQ check to be analyzed with each batch for samples from SC (13.3). Added requirement for insoluble matrix spike for solid samples (12.4) and post digestion spike for samples with an MS outside control limits (19.5). Updated calibration verification requirements to ICV after calibration and CCV prior to sample analysis. Added Appendix A listing project-specific optional corrective actions for MS failures.	Shelley Bourgeois

Appendix A – Corrective Actions for Matrix Spike Nonconformances

There are many corrective actions listed in method EPA 3060A for QC failure of either a soluble or insoluble matrix spike for solid sample preparation and analysis. GCAL's standard procedure for matrix spike failure is to analyze a post-digestion spike. If the LCS recovery is within control limits and the soluble matrix spike, insoluble matrix spike, and/or post digestion spike are outside control limits, the data will be reported with a case narrative unless additional corrective action is required on a project-specific basis.

Optional corrective actions include analysis of Oxidation Reduction Potential (ORP) and pH. A 20 g aliquot of sample will be mixed with 20 mL of deionized water and the 1:1 extract will be analyzed for ORP and pH. The Eh and pH values will be reported to the client for determination of the sample's reducing or oxidizing nature (see Figure 2, method 3060A).

Additional analysis of reducing agents, such as ferrous iron, sulfide, and TOC may be performed if requested by the client, and will be reported per the appropriate GCAL SOP.

HACH STANDARD IDs (1)

Date: _____
Analyst: _____
HBN: _____
Batch: _____

Test: _____

Calibration Standard Lot: _____

Calibration Std. Exp: _____

Calibration ID: _____

Calibration Date: _____

Calibration Exp: _____

Secondary Reviewer/Date: _____

Note: Calibration expires 1 year from calibration date or on the calibration standard expiration date, whichever is sooner.

SURFACTANTS - SM 5540 C-2011

CCV Standard ID (made daily) _____

ICV Standard ID (made daily) _____

Prep Batch HBN _____

COLOR - NCASI Tech. Bulletin 253

CCV (LCS) Standard ID (made daily) _____

ICV Standard ID (made daily) _____

pH meter _____

0.45 um Filter Lot # _____

SILICA - SM 4500-SiO_2 C-2011

CCV (LCS) Standard ID (made daily) _____

ICV Standard ID (made daily) _____

Molybdate Powder Pillow Lot #/Exp. Date _____

Citric Acid Powder Pillow Lot #/Exp. Date _____

Acid Reagent Powder Pillow Lot #/Exp. Date _____

ORTHOPHOSPHATE - SM 4500-P E-2011

CCV (LCS) Standard ID (made daily) _____

ICV Standard ID (made daily) _____

PhosVer3 Powder Pillow Lot #/Exp. Date _____

HEXAVALENT CHROMIUM - EPA 7196A / SM 3500-Cr B-2011

CCV Standard ID (made daily) _____

ICV Standard ID (made daily) _____

ChromaVer3 Powder Pillow Lot #/Exp. Date _____

pH meter _____

0.45 um Filter Lot # _____

Prep Batch HBN _____

SULFATE - EPA 9038 / SM 4500-SO_4 E-2011

CCV (LCS) Standard ID (made daily) _____

ICV Standard ID (made daily) _____

Conditioning Reagent Lot #/Exp. Date _____

Barium Chloride Lot #/Exp. Date _____

GCAL ANALYTICAL LABORATORIES, LLC

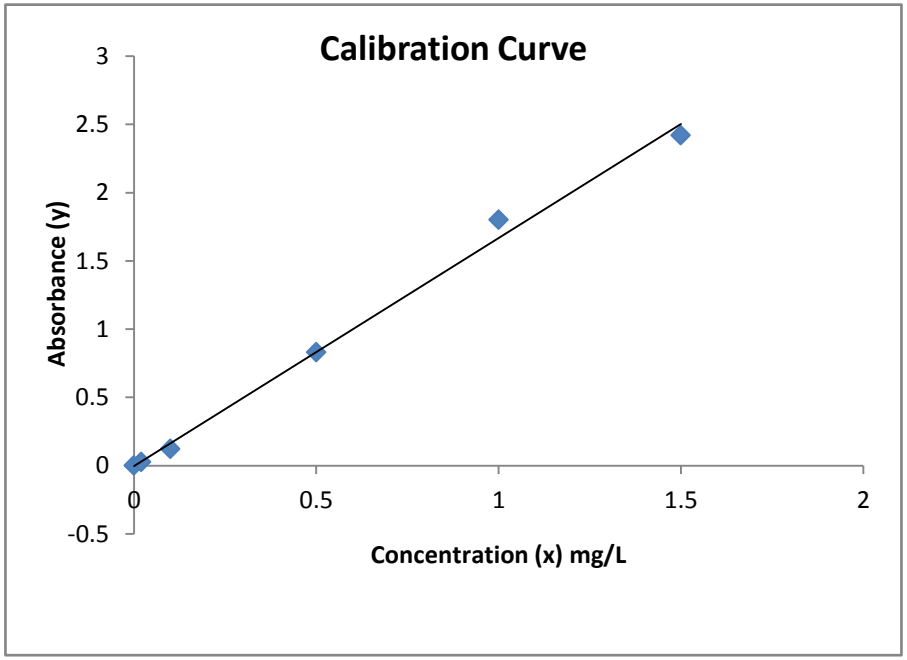
HACH Initial Calibration

Method: _____
Instrument: _____
Analyst: _____
Date: _____
Expiration: _____
Secondary Review: _____

Curve Type: Linear Least Square Regression
Origin: Not Forced
Weighting: None
Formula: y = mx + b

Data		
True Concentration (x)	Absorbance (y)	%Recovery
0	0.001	NA
0.02	0.026	83
0.1	0.123	75
0.5	0.831	100
1	1.8	108
1.5	2.419	97
		#DIV/0!
		#DIV/0!
		#DIV/0!

* Denotes outside of control limits



Statistics	
Slope - m	1.670
Intercept - b	-0.002
Correlation - r	0.998

Control Limits
Recovery - Lowest Non-Zero Pt 50-150%
Correlation Coefficient - r ≥ 0.995

Revision 1
11/18/13

Appendix C
Screening Levels and/or Criterion

APPENDIX C
Soil Screening Levels and Criterion

Chemical	CAS No	Analytical Method	RSL Industrial	RSL Residential	RSL PG SSL RISK	RSL PG SSL MCL	R4 ECO SOIL	NOAA SOIL
VOCs (µg/kg)								
1,1,1,2-Tetrachloroethane	630-20-6	8260B	9300	1900	0.19	NE	NE	225000
1,1,1-Trichloroethane	71-55-6	8260B	3800000	870000	260	70	NE	70 D
1,1,2,2-Tetrachloroethane	79-34-5	8260B	2800	560	0.026	NE	NE	127
1,1,2-Trichloroethane	79-00-5	8260B	680	160	0.013	1.6	NE	400 D
1,1-Dichloroethane	75-34-3	8260B	17000	3300	0.68	NE	NE	20 D
1,1-Dichloroethene	75-35-4	8260B	110000	24000	9.3	2.5	NE	100 D
1,1-Dichloropropene	563-58-6	8260B	NE	NE	NE	NE	NE	NE
1,2,3-Trichlorobenzene	87-61-6	8260B	49000	4900	1.5	NE	NE	30 D
1,2,3-Trichloropropane	96-18-4	8260B	95	5	0.00028	NE	NE	3360
1,2,4-Trichlorobenzene	120-82-1	8260B	27000	6200	1.1	200	NE	30 D
1,2,4-Trimethylbenzene	95-63-6	8260B	26000	6200	2.1	NE	NE	NE
1,2-Dibromo-3-chloropropane	96-12-8	8260B	69	5.4	0.00014	0.086	NE	35.2
1,2-Dibromoethane	106-93-4	8260B	170	34	0.0018	0.014	NE	1230
1,2-Dichlorobenzene	95-50-1	8260B	980000	190000	27	580	NE	30 D
1,2-Dichloroethane	107-06-2	8260B	2200	430	0.042	1.4	400	--
1,2-Dichloropropane	78-87-5	8260B	4700	940	0.13	1.7	700000	--
1,3,5-Trimethylbenzene	108-67-8	8260B	1000000	78000	12	NE	NE	NE
1,3-Dichlorobenzene	541-73-1	8260B	NE	NE	NE	NE	NE	30 D
1,3-Dichloropropane	142-28-9	8260B	2000000	160000	9.9	NE	NE	NE
1,4-Dichlorobenzene	106-46-7	8260B	12000	2400	0.4	72	NE	30 D
2,2-Dichloropropane	594-20-7	8260B	NE	NE	NE	NE	NE	NE
2-Butanone	78-93-3	8260B	20000000	2800000	100	NE	NE	89600
2-Chlorotoluene	95-49-8	8260B	2000000	160000	17	NE	NE	NE
2-Hexanone	591-78-6	8260B	140000	21000	0.79	NE	NE	12600
4-Chlorotoluene	106-43-4	8260B	2000000	160000	18	NE	NE	NE
4-Methyl-2-pentanone	108-10-1	8260B	5300000	530000	23	NE	NE	443000
Acetone	67-64-1	8260B	630000000	6100000	240	NE	NE	2500
Benzene	71-43-2	8260B	5400	1100	0.2	2.6	50	--
Bromobenzene	108-86-1	8260B	180000	30000	3.6	NE	NE	NE
Bromochloromethane	74-97-5	8260B	68000	16000	2.1	NE	NE	NE
Bromodichloromethane	75-27-4	8260B	1400	270	0.032	22	NE	540
Bromoform	75-25-2	8260B	2200000	62000	2.1	21	NE	15900
Bromomethane	74-83-9	8260B	3200	730	0.18	NE	NE	NE
Carbon disulfide	75-15-0	8260B	370000	82000	21	NE	NE	94.1
Carbon tetrachloride	56-23-5	8260B	3000	610	0.15	1.9	1000000	--
Chlorobenzene	108-90-7	8260B	140000	29000	4.9	68	50	--
Chloroethane	75-00-3	8260B	6100000	1500000	590	NE	NE	NE
Chloroform	67-66-3	8260B	1500	290	0.053	22	1	--
Chloromethane	74-87-3	8260B	50000	12000	4.9	NE	NE	NE
cis-1,2-Dichloroethene	156-59-2	8260B	500000	16000	0.82	21	NE	200 D
cis-1,3-Dichloropropene	10061-01-5	8260B	NE	NE	NE	NE	NE	398
Dibromochloromethane	124-48-1	8260B	3300	680	0.039	21	NE	2050
Dibromomethane	74-95-3	8260B	11000	2800	0.19	NE	NE	65000
Dichlorodifluoromethane	75-71-8	8260B	40000	9700	30	NE	NE	39500
Ethylbenzene	100-41-4	8260B	27000	5400	1.5	780	50	--
Hexachlorobutadiene	87-68-3	8260B	22000	6100	0.5	NE	NE	39.8
Isopropylbenzene	98-82-8	8260B	1100000	210000	64	NE	NE	NE
m- and p-Xylene	108-38-3/106-42	8260B	250000	59000	18	NE	NE	100
Methyl Tert-Butyl Ether	1634-04-4	8260B	220000	43000	2.8	NE	NE	NE
Methylene chloride	75-09-2	8260B	310000	36000	2.2	1.3	NE	400 D
n-Butylbenzene	104-51-8	8260B	5100000	390000	250	NE	NE	NE
Propylbenzene	103-65-1	8260B	2100000	340000	99	NE	NE	NE
o-Xylene	95-47-6	8260B	300000	69000	19	NE	NE	100

APPENDIX C
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Chemical	CAS No	Analytical Method	RSL Industrial	RSL Residential	RSL PG SSL RISK	RSL PG SSL MCL	R4 ECO SOIL	NOAA SOIL
4-Isopropyltoluene	99-87-6	8260B	NE	NE	NE	NE	NE	NE
sec-Butylbenzene	135-98-8	8260B	10000000	780000	460	NE	NE	NE
Styrene	100-42-5	8260B	3600000	630000	120	110	100	--
tert-Butylbenzene	98-06-6	8260B	10000000	780000	110	NE	NE	NE
Tetrachloroethene	127-18-4	8260B	41000	8600	1.6	2.3	10	--
Toluene	108-88-3	8260B	4500000	500000	59	690	50	--
trans-1,2-Dichloroethene	156-60-5	8260B	69000	15000	2.5	29	NE	200 D
trans-1,3-Dichloropropene	10061-02-6	8260B	NE	NE	NE	NE	NE	398
Trichloroethene	79-01-6	8260B	2000	440	0.092	1.8	NE	100 D
Trichlorofluoromethane	75-69-4	8260B	340000	49000	69	NE	NE	16400
Vinyl chloride	75-01-4	8260B	1700	60	0.0053	0.69	10	--
SVOCs (µg/kg)								
1,2,4-Trichlorobenzene	120-82-1	8270D	27000	6200	1.1	200	NE	30 D
1,2-Diphenylhydrazine	122-66-7	8270D	2200	610	0.22	NE	NE	NE
2,4,5-Trichlorophenol	95-95-4	8270D	6200000	610000	330	NE	400	--
2,4,6-Trichlorophenol	88-06-2	8270D	62000	6100	3.4	NE	10000	--
2,4-Dichlorophenol	120-83-2	8270D	180000	18000	4.1	NE	NE	10 D
2,4-Dimethylphenol	105-67-9	8270D	1200000	120000	32	NE	NE	10 v
2,4-Dinitrophenol	51-28-5	8270D	120000	12000	3.4	NE	NE	60.9
2,4-Dinitrotoluene	121-14-2	8270D	5500	1600	0.28	NE	NE	1280
2,6-Dichlorophenol	87-65-0	8270D	NE	NE	NE	NE	NE	10 D
2,6-Dinitrotoluene	606-20-2	8270D	1200	330	0.058	NE	NE	32.8
2-Chloronaphthalene	91-58-7	8270D	8200000	630000	290	NE	NE	12.2
2-Chlorophenol	95-57-8	8270D	510000	39000	5.7	NE	NE	10 D
2-Methylphenol	95-48-7	8270D	3100000	310000	5.8	NE	NE	50 D
2-Nitroaniline	88-74-4	8270D	600000	61000	6.2	NE	NE	74100
2-Nitrophenol	88-75-5	8270D	NE	NE	NE	NE	NE	1600
3,3-Dichlorobenzidine	91-94-1	8270D	3800	1100	0.71	NE	NE	646
3- and 4-Methylphenol	108-39-4/106-44	8270D	3100000	310000	57	NE	NE	50 D
3-Nitroaniline	99-09-2	8270D	NE	NE	NE	NE	NE	NE
4,6-Dinitro-2-methylphenol	534-52-1	8270D	4900	490	0.2	NE	NE	NE
4-Bromophenyl-phenylether	101-55-3	8270D	NE	NE	NE	NE	NE	NE
4-Chloro-3-methylphenol	59-50-7	8270D	6200000	610000	130	NE	NE	7950
4-Chloroaniline	106-47-8	8270D	8600	2400	0.13	NE	NE	30 D
4-Chlorophenyl-phenylether	7005-72-3	8270D	NE	NE	NE	NE	NE	NE
4-Nitroaniline	100-01-6	8270D	86000	24000	1.4	NE	NE	NE
4-Nitrophenol	100-02-7	8270D	NE	NE	NE	NE	700	--
Benzidine	92-87-5	8270D	7.5	0.5	0.00024	NE	NE	NA
Benzoic acid	65-85-0	8270D	250000000	24000000	1400	NE	NE	NA
Benzyl alcohol	100-51-6	8270D	6200000	610000	37	NE	NE	65800
Bis(2-chloroethoxy)methane	111-91-1	8270D	180000	18000	1.1	NE	NE	302
Bis(2-chloroethyl)ether	111-44-4	8270D	1000	210	0.0031	NE	NE	23700
Bis(2-chloroisopropyl)ether	39638-32-9	8270D	NE	NE	NE	NE	NE	NE
Bis(2-ethylhexyl)phthalate	117-81-7	8270D	120000	35000	1100	1400	NE	100 D
Butylbenzylphthalate	85-68-7	8270D	910000	260000	200	NE	NE	100 D
Carbazole	86-74-8	8270D	NE	NE	NE	NE	NE	NE
Dibenzofuran	132-64-9	8270D	100000	7800	11	NE	NE	NE
Diethylphthalate	84-66-2	8270D	49000000	4900000	470	NE	100000	--
Dimethyl phthalate	131-11-3	8270D	NE	NE	NE	NE	200000	--
Di-n-butylphthalate	84-74-2	8270D	6200000	610000	170	NE	200000	--
Di-n-octylphthalate	117-84-0	8270D	620000	61000	4400	NE	NE	100 D
Hexachlorobenzene	118-74-1	8270D	1100	300	0.53	13	2.5	--
Hexachloroethane	67-72-1	8270D	43000	4300	0.31	NE	NE	596
Isophorone	78-59-1	8270D	1800000	510000	22	NE	NE	139000

APPENDIX C
Soil Screening Levels and Criterion

Chemical	CAS No	Analytical Method	RSL Industrial	RSL Residential	RSL PG SSL RISK	RSL PG SSL MCL	R4 ECO SOIL	NOAA SOIL
Nitrobenzene	98-95-3	8270D	24000	4800	0.079	NE	40000	--
N-Nitrosodinpropylamine	621-64-7	8270D	250	69	0.007	NE	NE	544
N-Nitrosodiphenylamine	86-30-6	8270D	350000	99000	57	NE	20000	--
N-Nitrosodimethylamine	62-75-9	8270D	34	2.3	0.0001	NE	NE	0.0321
Pentachlorophenol	87-86-5	8270D	2700	890	0.36	10	2	--
Phenol	108-95-2	8270D	18000000	1800000	260	NE	50	--
2-Methylnaphthalene	91-57-6	8270D SIM	220000	23000	14	NE	NE	3240
Acenaphthene	83-32-9	8270D SIM	3300000	340000	410	NE	NE	20000
Acenaphthylene	208-96-8	8270D SIM	NE	NE	NE	NE	NE	682000
Anthracene	120-12-7	8270D SIM	17000000	1700000	4200	NE	100	--
Benzo[a]anthracene	56-55-3	8270D SIM	2100	150	10	NE	NE	5210
Benzo[a]pyrene	50-32-8	8270D SIM	210	15	3.5	240	100	--
Benzo[b]fluoranthene	205-99-2	8270D SIM	2100	150	35	NE	NE	59800
Benzo[g,h,i]perylene	191-24-2	8270D SIM	NE	NE	NE	NE	NE	119000
Benzo[k]fluoranthene	207-08-9	8270D SIM	21000	1500	350	NE	NE	148000
Chrysene	218-01-9	8270D SIM	210000	15000	1100	NE	NE	4730
Dibenz[a,h]anthracene	53-70-3	8270D SIM	210	15	11	NE	NE	18400
Fluoranthene	206-44-0	8270D SIM	2200000	230000	7000	NE	100	--
Fluorene	86-73-7	8270D SIM	2200000	230000	400	NE	30000	--
Indeno[1,2,3-cd]pyrene	193-39-5	8270D SIM	2100	150	200	NE	NE	109000
Naphthalene	91-20-3	8270D SIM	18000	3600	0.47	NE	100	--
Phenanthrene	85-01-8	8270D SIM	NE	NE	NE	NE	100	--
Pyrene	129-00-0	8270D SIM	1700000	170000	950	NE	100	--
Pesticides (µg/kg)								
4,4-DDD	72-54-8	8081B	7200	2000	6.4	NE	2.5	--
4,4-DDE	72-55-9	8081B	5100	1400	46	NE	2.5	--
4,4-DDT	50-29-3	8081B	7000	1700	67	NE	2.5	--
Aldrin	309-00-2	8081B	100	29	0.65	NE	2.5	--
alpha-BHC	319-84-6	8081B	270	77	0.036	NE	NE	3 D
alpha-Chlordane	5103-71-9	8081B	NE	NE	NE	NE	NE	0.03 D
beta-BHC	319-85-7	8081B	960	270	0.13	NE	NE	9 D
delta-BHC	319-86-8	8081B	NE	NE	NE	NE	NE	10 D
Dieldrin	60-57-1	8081B	110	30	0.061	NE	0.5	--
Endosulfan I	959-98-8	8081B	NE	NE	NE	NE	NE	0.01 D
Endosulfan II	33213-65-9	8081B	NE	NE	NE	NE	NE	0.01 D
Endosulfan sulfate	1031-07-8	8081B	NE	NE	NE	NE	NE	35.8
Endrin	72-20-8	8081B	18000	1800	6.8	81	1	--
Endrin aldehyde	7421-93-4	8081B	NE	NE	NE	NE	NE	10.5
Endrin ketone	53494-70-5	8081B	NE	NE	NE	NE	NE	NE
Lindane	58-89-9	8081B	2100	520	0.21	1.2	0.05	--
gamma-Chlordane	5566-34-7	8081B	NE	NE	NE	NE	NE	0.03
Heptachlor	76-44-8	8081B	380	110	0.14	33	NE	0.7 D
Heptachlor epoxide	1024-57-3	8081B	190	53	0.068	4.1	NE	0.0002 D
Methoxychlor	72-43-5	8081B	310000	31000	150	2200	NE	19.9
PCBs (µg/kg)								
Aroclor-1016	12674-11-2	8082A	3700	390	10	NE	20	--
Aroclor-1221	11104-28-2	8082A	540	140	0.069	NE	20	--
Aroclor-1232	11141-16-5	8082A	540	140	0.069	NE	20	--
Aroclor-1242	53469-21-9	8082A	740	220	5.3	NE	20	--
Aroclor-1248	12672-29-6	8082A	740	220	5.2	NE	20	--
Aroclor-1254	11097-69-1	8082A	740	110	8.2	NE	20	--
Aroclor-1260	11096-82-5	8082A	740	220	24	NE	20	--
Aroclor-1262	37324-23-5	8082A	NE	NE	NE	NE	20	--
Aroclor-1268	11100-14-4	8082A	NE	NE	NE	NE	20	--

APPENDIX C
Soil Screening Levels and Criterion

Chemical	CAS No	Analytical Method	RSL Industrial	RSL Residential	RSL PG SSL RISK	RSL PG SSL MCL	R4 ECO SOIL	NOAA SOIL
Metals (µg/kg)								
Aluminum	7429-90-5	6020A	99000000	7700000	2300000	NE	50000	--
Antimony	7440-36-0	6020A	41000	3100	27	270	3500	--
Arsenic	7440-38-2	6020A	2400	610	1.3	290	10000	--
Barium	7440-39-3	6020A	19000000	1500000	12000	82000	165000	--
Beryllium	7440-41-7	6020A	200000	16000	1300	3200	1100	--
Cadmium	7440-43-9	6020A	NE	NE	52	380	1600	--
Calcium	7440-70-2	6020A	NE	NE	NE	NE	NE	NE
Chromium, Total	7440-47-3	6020A	NE	NE	NE	180000000	400	--
Chromium, Hexavalent	18540-29-9	7196A	5600	290	0.59	NE		
Cobalt	7440-48-4	6020A	30000	2300	21	NE	20000	--
Copper	7440-50-8	6020A	4100000	310000	2200	46000	40000	--
Iron	7439-89-6	6020A	72000000	5500000	27000	NE	200000	--
Lead	7439-92-1	6020A	800000	400000	NE	14000	50000	--
Magnesium	7439-95-4	6020A	NE	NE	NE	NE	NE	NE
Manganese	7439-96-5	6020A	2300000	180000	2100	NE	100000	--
Mercury	7439-97-6	7470/7471A	4300	1000	3.3	100	NE	
Molybdenum	7439-98-7	6020A	510000	39000	160	NE	2000	--
Nickel	7440-02-0	6020A	2000000	150000	2000	NE	30000	--
Potassium	7440-09-7	6020A	NE	NE	NE	NE	NE	NE
Selenium	7782-49-2	6020A	510000	39000	40	260	810	--
Silver	7440-22-4	6020A	510000	39000	60	NE	2000	--
Sodium	7440-23-5	6020A	NE	NE	NE	NE	NE	NE
Thallium	7440-28-0	6020A	1000	78	1.1	140	1000	--
Vanadium	7440-62-2	6020A	510000	39000	6300	NE	2000	--
Zinc	7440-66-6	6020A	31000000	2300000	29000	NE	50000	--

Notes:

VOCs	=	Volatile organic compounds
SVOCs	=	Semi-volatile organic compounds
PCBs	=	Polychlorinated biphenyl compounds
CAS	=	Chemical Abstracts Service
RSL	=	Regional Screening Levels
NE	=	Not established
µg/kg	=	Microgram per kilogram
U.S. EPA	=	U.S. Environmental Protection Agency
NOAA	=	National Oceanic and Atmosphere Administration, Lowest Value from Screening Quick reference Tables
RSL Residential	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites — Residential (U.S. EPA, Nov 2013)
RSL Industrial	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites — Industrial (U.S. EPA, Nov 2013)
RSL PG SSL RISK	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites — Risk-based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov 2013)
RSL PG SSL MCL	=	U.S. EPA Regional Screening Levels for Chemical Contaminants at Superfund Sites — Maximum Contaminant Level-Based Soil Screening Levels for Protection of Groundwater (U.S. EPA, Nov
R4 Eco Soil	=	U.S. EPA Region 4 Ecological Soil Screening Level (U.S. EPA, 2001).