

SAP Worksheet #1—Title and Approval Page

**Amended Final
Sampling and Analysis Plan
Pilot Test at Solid Waste Management Units 54 and 55
Naval Activity Puerto Rico
Ceiba, Puerto Rico
January 2011**

Prepared for:



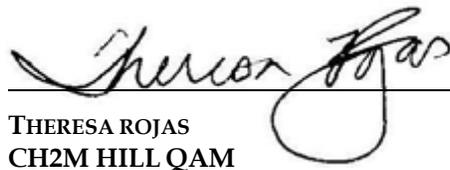
Prepared by:



Prepared under:

Small Business Remedial Action Contract N62470-08-D-1006
Task Order No. JM04

REVIEW SIGNATURE:



THERESA ROJAS
CH2M HILL QAM

OTHER APPROVAL SIGNATURES:



MARK E. DAVIDSON
BRAC PMO SE - REMEDIAL PROJECT MANAGER

APPROVAL SIGNATURE:



JON TUCKER
NAVFAC ATLANTIC - CHEMIST/QA OFFICER

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Executive Summary

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Naval Facilities Engineering Command Southeast (NAVFAC SE) to implement the preferred corrective measure technologies, as indicated in the *Final Corrective Measures Study [CMS] Final Report for SWMUs 54 and 55* (Baker Environmental, Inc. [Baker], 2005), to address groundwater contamination at Solid Waste Management Units (SWMUs) 54 and 55 located at Naval Activity Puerto Rico (NAPR) Ceiba, Puerto Rico (Figures 1 and 2 in Appendix A). This work is being performed under Contract Number N62470-08-D-1006, Task Order JM04.

In February 2002, a Final Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) was conducted at SWMU 54 to evaluate soil and groundwater contamination identified during a site characterization investigation. According to the RFI report, no further action was required to mitigate surface soil or subsurface soil (Baker, 2003). However, trichloroethene (TCE) contamination in groundwater was identified and delineated as a result of the RFI. A plume of benzene was also delineated in the vicinity of Bairoko Street. Because of the relatively high levels of benzene, it was recommended that a CMS be developed to determine remedial alternatives for contaminants in the groundwater at this site. According to the final CMS, only two contaminants in groundwater exceeded their respective human health based corrective action objectives (CAOs): TCE east of Bairoko Street and benzene west of Bairoko Street (Baker, 2005). The TCE CAO is 22 micrograms per liter ($\mu\text{g}/\text{L}$), while the benzene CAO is 550 $\mu\text{g}/\text{L}$.

Between July 2009 and April 2010, AGVIQ-CH2M HILL conducted an additional investigation at SWMU 54 to evaluate groundwater contamination identified in the final CMS (Baker, 2005). The investigation confirmed two benzene plumes exist: one in a shallow zone primarily on the west side of Bairoko Street and one in a deep zone on the east side of Bairoko Street. In addition, the investigation identified a previously unknown area of shallow benzene contamination on the southeast side of Bairoko Street (AGVIQ-CH2M HILL, 2010a). The investigation also showed that the TCE plume shifted slightly to the south (AGVIQ-CH2M HILL, 2010b). According to the final CMS, environmental investigations conducted at SWMU 55 concluded TCE in groundwater is the only compound in excess of the CAO of 22 $\mu\text{g}/\text{L}$ at SWMU 55 (Baker, 2005). A TCE plume delineation and source investigation was performed in September 2003 (Baker, 2004). During this investigation, the maximum TCE concentration in groundwater was measured at monitoring well 7MW7 at 1,800 $\mu\text{g}/\text{L}$.

Between August 2009 and April 2010, AGVIQ-CH2M HILL conducted an additional investigation to evaluate the groundwater contamination identified in the final CMS (Baker, 2005). During this investigation, the maximum TCE concentration in groundwater was measured at injection well 55IW01 at 33,600 $\mu\text{g}/\text{L}$ (AGVIQ-CH2M HILL, 2010c). This document is the Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) for the work to be performed at SWMUs 54 and 55 to remediate groundwater impacted by volatile organic compounds. Remediation activities to be conducted at SWMUs 54 and 55 include:

- Pilot-scale testing will be conducted to evaluate in situ biodegradation (ISB) via enhanced reductive dechlorination to remediate TCE in groundwater at SWMU 54. ISB was selected to enhance the natural processes already occurring and to reduce the time required to achieve CAOs.

- Pilot-scale testing will be conducted to evaluate air sparging to remediate the benzene in groundwater at SWMU 54.
- Pilot-scale testing will be conducted to evaluate in situ chemical oxidation to remediate TCE in groundwater at SMWU 55.

SAP Format

This SAP has been prepared in accordance with the UFP for Quality Assurance Project Plans (UFP-QAPP) (U.S. Environmental Protection Agency [EPA], 2005) and the EPA Guidance for QAPPs, EPA QA/G-5, QAMS (EPA, 2002), and contains the 37 worksheets identified in Part 2A. It also contains appendices that support the information presented in the worksheets. Figures are presented in Appendix A.

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A Figures

- 1 SWMU 54 Location
- 2 SWMU 55 Location
- 3 Site Location Map, SWMU 54
- 4 Injection and Monitoring Well Locations - TCE Plume, SWMU 54
- 5 Injection and Monitoring Well Locations - Benzene Plume, SWMU 54
- 6 Location of Monitoring and Injection Wells, SWMU 55

B Standard Operating Procedures

C Standard Label and Chain-of-Custody Record

D Data Validation Checklists for SW-846 Methods, Level IV Data Package Deliverables, and Data Reporting Form

E Laboratory Quality Assurance Program Plan, Laboratory Audit Checklists, and Custody Seal

F Technical Memorandums for SWMUs 54 and 55

Tables

- 1 Sample Collection Schedule

Acronyms and Abbreviations

°C	degree Celsius
%R	percent recovery
AGVIQ-CH2M HILL	AGVIQ-CH2M HILL Joint Venture III
AS	air sparge
ASAP	as soon as possible
Baker	Baker Environmental, Inc.
Bgs	below ground surface
BRAC PMO SE	Base Realignment and Closure Program Management Office Southeast
CA	Corrective Action
CAO	Corrective Action Objective
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CMS	Corrective Measures Study
COC	contaminant of concern
COD	chemical oxygen demand
CPR	cardiopulmonary resuscitation
CV	calibration verification
DCE	dichloroethene
DHE	dehalocoides ethennogenes
DO	dissolved oxygen
DOD	U.S. Department of Defense
DQE	data quality evaluation
DQI	data quality indicator
DRMO	Defense Reutilization and Marketing Office
EIS	Environmental Information Specialist
EPA	U.S. Environmental Protection Agency
ERD	enhanced reductive dechlorination
ERP	Environmental Restoration Program
EVO	emulsified vegetable oil
FID	flame ionized detector
FTL	Field Team Leader
GCAL	Gulf Coast Analytical Laboratories, Inc.
GC/MS	gas chromatography/mass spectrometer
g/L	gram per liter
gpm	gallon per minute
HSA	hollow stem auger
HSP	Health and Safety Plan
HSWA	Hazardous and Solid Waste Amendments
HAZWOPER	Hazardous Waste Operations and Emergency Response
ICAL	initial calibration
ICV	initial calibration verification
ID	identification

IRP	Installation Restoration Program
ISB	in situ biodegradation
ISCO	in situ chemical oxidation
ITRC	Interstate Technology Regulatory Council
KMnO ₄	potassium permanganate
LCS	laboratory control sample
LDO	luminescent dissolved oxygen
LIMS	Laboratory Information Management System
µg/L	microgram per liter
MEE	methane, ethane, ethene
mg/L	milligram per liter
MNA	monitored natural attenuation
MnO ₄ ⁻	permanganate
MS	matrix spike
MSD	matrix spike duplicate
NaMnO ₄	sodium permanganate
NAPR	Naval Activity Puerto Rico
NAVFAC SE	Naval Facilities Engineering Command Southeast
Navy	U.S. Department of Navy
NEX	Naval Exchange
NFESC	Naval Facilities Engineering Service Center
NIRIS	Navy Installation Restoration Information System
NSRR	Naval Station Roosevelt Roads
ORP	oxidation-reduction potential
PAL	Project Action Limit
PDF	portable document format
PDS	post-digestion spike
PM	Project Manager
POC	point of contact
PREQB	Puerto Rico Environmental Quality Board
PQO	Project Quality Objective
PWR	partially weathered rock
QA	quality assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
ROI	radius of influence
RPD	relative percent difference
RPM	Remedial Project Manager
RSD	relative standard deviation
SAP	Sampling and Analysis Plan
SOP	standard operating procedure
SRM	standard reference material
SSC	Site Safety Coordinator
SWMU	solid waste management unit

TBD	to be determined
TCE	Trichloroethene
TCLP	Toxicity Characteristic Leaching Procedure
TOC	total organic carbon
TOD	total oxidant demand
TWFF	Tow Way Fuel Farm
UFP	Uniform Federal Policy
UST	underground storage tank
VOC	volatile organic compound

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SAP Worksheet #2—SAP Identifying Information

Site Name/Number: NAPR, SWMUs 54 and 55
Operable Unit: 2
Contractor Name: AGVIQ-CH2M HILL
Contract Number: N62470-08-D-1006-JM04
Contract Title: Small Business Remedial Action Contract

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) (U.S. Environmental Protection Agency [EPA], 2005) and EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (EPA, 2002).
2. Regulatory program: Resource Conservation and Recovery Act (RCRA)
3. This SAP is a project-specific SAP.
4. Dates of scoping sessions:

Scoping Session	Date
Site visit – Ceiba, Puerto Rico	December 17, 2008
Site visit – Ceiba, Puerto Rico	January 19 – 23, 2009
Technical Approach Meeting	February 4, 2009

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

Title	Date
Baker Environmental, Inc. (Baker). 2005. <i>Final Corrective Measures Study Final Report for SWMUs 54 and 55</i> . Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division.	August 2005

6. Organizational partners (stakeholders) and connection with lead organization:
 - **EPA Region II** – Regulatory stakeholder overseeing RCRA Ceiba Environmental Restoration Program (ERP) implemented by lead organization
 - **Puerto Rico Environmental Quality Board (PREQB)** – Regulatory stakeholder overseeing RCRA Ceiba ERP implemented by lead organization
 - **U.S. Naval Facilities Engineering Command Southeast (NAVFAC SE)** – Performs remedial activities at specified sites at the Naval Activity Puerto Rico (NAPR).

7. Lead organization (see Worksheet #7 for detailed list of data users):

– **U.S. Department of Navy (Navy)**

8. The omitted SAP elements excluded and provide an explanation for their exclusion below:

Crosswalk table is excluded as all required information is provided in this SAP.

SAP Worksheet #3—Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number
David Criswell Mark E. Davidson	Base Realignment and Closure Program Management Office Southeast (BRAC PMO SE) Remedial Project Manager (RPM)/Lead Navy Point of Contact (POC)	Navy	(843) 743-2130 (843) 743-2124	david.criswell@navy.mil mark.e.davidson@navy.mil	
To be determined (TBD)	Contracting Officer	Navy	TBD	TBD	
TBD	Librarian and Records Manager/Final document archiving	Navy	TBD	TBD	
Amy Wolff (will distribute to the Program Management Office)	Program Assistant/Document Manager	AGVIQ-CH2M HILL	(678) 530-4393	Amy.Wolff@ch2m.com	
Doug Downey	Senior Technical Consultant	AGVIQ-CH2M HILL	(303) 674-6547	Doug.downey@ch2m.com	
Tom Beisel	Project Manager (PM)	AGVIQ-CH2M HILL	(678) 530-4033	Tom.beisel@ch2m.com	
Camden Robinson	Project Chemist and Data Validator	AGVIQ-CH2M HILL	(678) 530-4292	Camden.robinson@ch2m.com	
Thomas Kessler	Senior Geologist	AGVIQ-CH2M HILL	(678) 530-4197	Thomas.kessler@ch2m.com	
Elizabeth Martin	Laboratory Project Manager	Gulf Coast Analytical Laboratories, Inc. (GCAL)	(225) 769-4900, ext. 308	elizabeth.martin@gcal.com	

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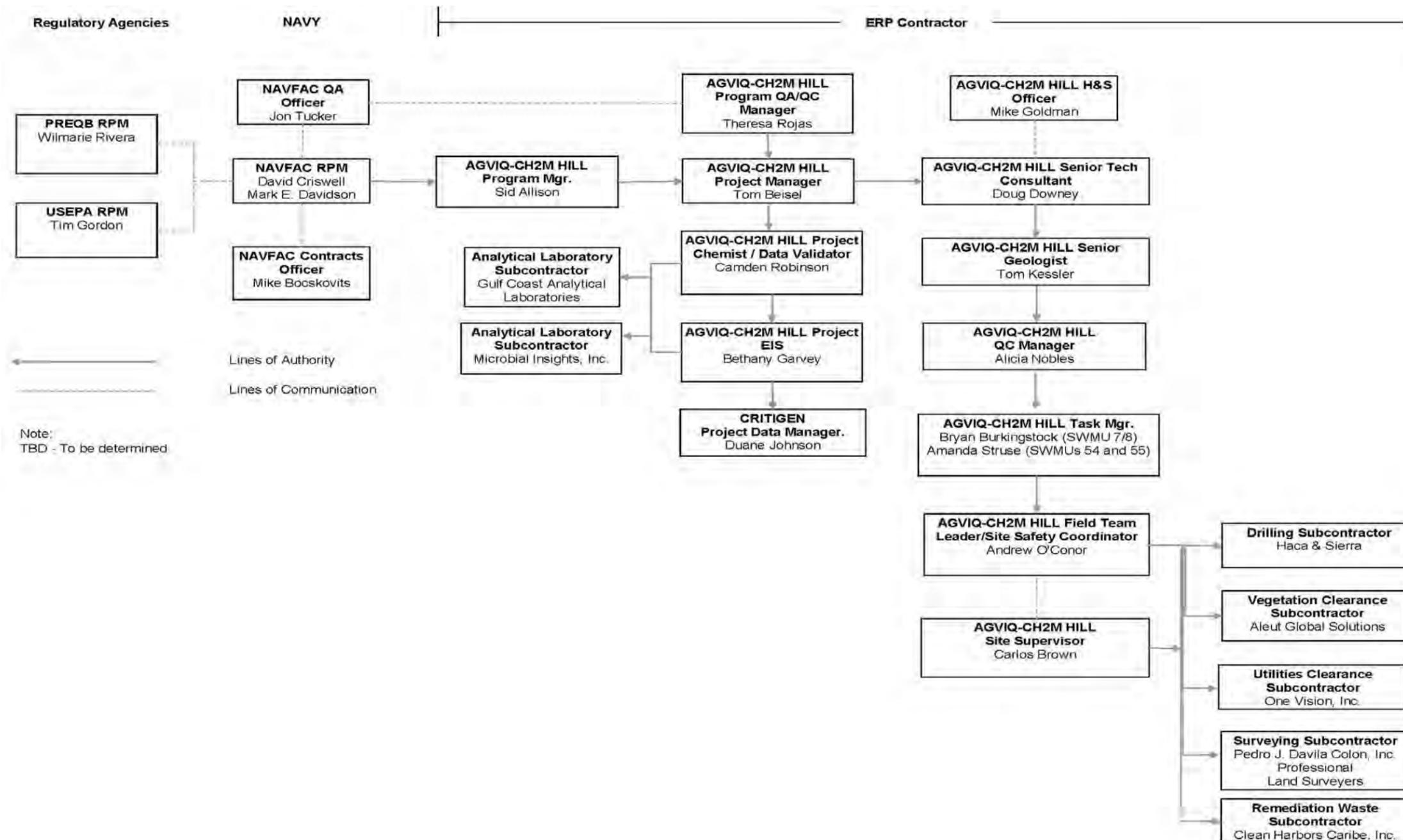
SAP Worksheet #4—Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number	Signature/E-mail Receipt	SAP Section Reviewed	Date SAP Read
David Criswell Mark E. Davidson	BRAC PMO SE RPM/Lead Navy POC	(843) 743-2130 (843) 743-2124			
Tim Gordon	EPA/Ceiba RPM/Regulatory POC	(212) 637-4167			
Wilmarie Rivera	PREQB/Ceiba RPM/Regulatory POC	(787) 767-8181, ext. 6141			
Theresa Rojas	AGVIQ-CH2M HILL/Program Quality Assurance (QA) Manager and Quality Control (QC) Manager/SAP Review	(678) 530-4297			
Camden Robinson	AGVIQ-CH2M HILL/Navy Program Chemist/SAP Review	(678) 530-4292			
Nancy Ballantyne	AGVIQ-CH2M HILL/Contractor Environmental Manager/Navy contractor primary POC	(720) 286-5561			
Bethany Garvey	AGVIQ-CH2M HILL/ Environmental Information Specialist (EIS)/	(678) 530-4124			
Duane Johnson	Critigen/Data Tracking and Management	(678) 530-4185			
Thomas Kessler Andrew O'Connor	AGVIQ-CH2M HILL/ Geologists/Field Team Leaders (FTLs)	(678) 530-4197 (843) 200-3825			
Elizabeth Martin	GCAL/Chemist/Laboratory PM	(225) 769-4900, ext. 308			
Anita Biernacki	Microbial Insights/Laboratory Contact	(865) 573-8188 ext. 108			

SAP Worksheet #4—Project Personnel Sign-Off Sheet (continued)

Name	Organization/Title/Role	Telephone Number	Signature/E-mail Receipt	SAP Section Reviewed	Date SAP Read
Andrew O'Connor Alicia Nobles	AGVIQ-CH2M HILL/Field Team	(843) 200-3825 (678) 530-4576			

SAP Worksheet #5—Project Organizational Chart



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SAP Worksheet #6—Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
Communication to/from Navy (e.g., submission of SAP for review; receipt of regulatory comments, etc.)	Navy RPM	David Criswell Mark E. Davidson	(843) 743-2130/ david.criswell@navy.mil (843) 743-2124/ mark.e.davidson@navy.mil	Primary POC for Navy (via e-mail, telephone, hardcopy, or in-person, as warranted); can delegate communication to other internal or external points of contact. Navy RPM to report any significant corrective actions (CAs) to the involved regulatory agencies, unless otherwise directed by the Navy RPM to AGVIQ-CH2M HILL PM.
Communication to/from EPA (e.g., receipt of SAP for review; submission of EPA comments)	EPA RPM	Tim Gordon	(212) 637-4167 Gordon.timothy@epa.gov	Primary POC for EPA (via e-mail, telephone, hardcopy, or in-person, as warranted); can delegate communication to other internal or external points of contact.
Communication to/from PREQB (e.g., receipt of SAP for review; submission of PREQB comments)	PREQB RPM	Wilmarie Rivera	(787) 767-8181, ext. 6141 Cell: (787) 365-8573	Primary POC for PREQB (via e-mail, telephone, hardcopy, or in-person, as warranted); can delegate communication to other internal or external points of contact.
Navy QA/QC input	Navy Quality Assurance Officer (QAO)	Jon Tucker	(757) 322-8288 Jonathan.tucker@navy.mil	Provides review comments to Navy contractor on pre-draft SAP via e-mail through Kevin Cloe. Provides overall Navy guidance via direct communication with Navy contractor QAO, as warranted.
Project administration and logistics Communication to/from Navy contractor (e.g., submission of SAP for review; receipt of regulatory comments, updates on project progress, communication of stakeholder expectations, etc.)	AGVIQ-CH2M HILL PM	Tom Beisel	(678) 530-4033	Direct communication (via e-mail, telephone, hardcopy, or in-person, as warranted) to/from Navy contractor project staff to ensure appropriate project implementation. Primary POC for Navy contractor (via e-mail, telephone, hardcopy, or in-person, as warranted); can delegate communication to other contractor staff, as appropriate.

SAP Worksheet #6—Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
Health and safety expectations and procedures	AGVIQ-CH2M HILL Health and Safety Officer	Mike Goldman	(865) 560-2908	Review of Health and Safety Plan (HSP). Direct communication (via e-mail, telephone, hardcopy, or in-person, as warranted) to/from AGVIQ-CH2M HILL project team to ensure implementation of appropriate health and safety procedures.
SAP changes in the field	AGVIQ-CH2M HILL FTL	Andrew O'Connor	(843) 200-3825	Documentation of deviations from work plan made in field logbooks and rationale for deviations; deviations made only with approval from AGVIQ-CH2M HILL PM. Deviations to the SAP will be reported within 1 week from the time the issue is identified.
Field CAs	AGVIQ-CH2M HILL FTL	Andrew O'Connor	(843) 200-3825	See Worksheets #32 and 32-1. Field CAs will be reported within 1 week from the time the issue is identified.
Daily Field Progress Reports	AGVIQ-CH2M HILL FTL	Andrew O'Connor	(843) 200-3825	FTL will e-mail or fax daily field progress reports to contractor PMs weekly; telephone communication with PMs on as-needed basis
Ensuring staff health and safety in the field	AGVIQ-CH2M HILL Site Safety Coordinator (SSC)	Andrew O'Connor	(843) 200-3825	Daily safety tailgates; daily observations; real-time discussions of observations and changes to be implemented with field staff.
Ensuring the project is meeting the requirements of this SAP and that any problems are corrected and communicated to the project administrator	AGVIQ-CH2M HILL QC Manager	Alicia Nobles	(678) 530-4576	Complete daily QC reports and submit to PM and project administrator.

SAP Worksheet #6—Communication Pathways (continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-mail	Procedure
Data tracking from collection through upload to database	AGVIQ-CH2M HILL EIS	Bethany Garvey	(678) 530-4124	EIS will track data from sample collection through upload to database, ensuring QAPP requirements are met by laboratory and field staff. Tracking involves receipt of electronic and hardcopy data from laboratory and data validator. EIS communicates with AGVIQ-CH2M HILL project chemist, laboratory PM, and data validator PM, as warranted, to ensure adherence to project analysis and validation requirements. EIS also coordinates data upload with contractor database manager.
Uploading project data and maintaining the database to ensure data are stored properly and can be retrieved by the EIS	Critigen Database Manager	Duane Johnson	(678) 530-4185	Once contractor chemist ensures data are appropriate for upload to database, EIS submits data electronically to contractor database manager, who uploads data to database.
Reporting lab data quality issues	Laboratory PM	Elizabeth Martin	(225) 769-4900 ext. 308	All QA/QC issues with project field samples will be reported by the lab to the EIS, Project Chemist, and Contractor QAO via e-mail within 2 business days.
Analytical CAs	AGVIQ-CH2M HILL Project Chemist	Camden Robinson	(770) 439-8363	See Worksheets #24, 25, and 28 for analytical CAs. Analytical CAs will be reported within 1 week from the time the issue is identified.
Validated data	Data Validator PM	Camden Robinson	(770) 439-8363	Data validator provides data validation reports (electronic and hardcopy) that provide the data qualifiers and associated explanations.
Release of analytical data for upload to database	AGVIQ-CH2M HILL Project Chemist	Camden Robinson	(770) 439-8363	Upon review of validated data to ensure adherence to project requirements, project chemist communicates via e-mail to EIS that data are ready for release (i.e., upload to database).

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SAP Worksheet #7—Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications ^a
David Criswell Mark E. Davidson	BRAC PMO SE RPM	Navy	Environmental restoration program activities implemented under this SAP.	
Jon Tucker	QAO	Navy	Navy review of SAP and QA input.	
Pedro Ruiz	Ceiba ERP Site Manager	Navy	On-island Navy liaison; provides logistical support for implementation of environmental restoration program activities under this SAP.	
Tom Beisel	PM	AGVIQ-CH2M HILL	Project administration; coordinates staffing; monitors project performance; directs and oversees project staff.	BS, Geology; over 18 years experience in project management, including staff supervision and project performance monitoring
Theresa Rojas	Program QA/QC Manager	AGVIQ-CH2M HILL	Oversees compliance with program and project-specific quality requirements.	BS, Chemistry; over 20 years experience in laboratory analysis, sampling, data validation, and field testing; over 15 years experience in construction quality management
Eric Burrell	Quality Control Plan Coordinator	AGVIQ-CH2M HILL	Oversees project-specific QC requirements.	BS, Civil Engineering; over 5 years experience in construction quality management
Camden Robinson	Project Chemist	AGVIQ-CH2M HILL	Establishes laboratory scope of work; ensures selected laboratory can meet project-required analytical protocol; primary communications with laboratory and data validator; performs data quality evaluation to determine availability of analytical data.	BA, Chemistry; over 5 years experience in chemistry, including laboratory analysis, sampling, data validation, and field testing
Bryan Burkingstock Amanda Struse	Task Manager	AGVIQ-CH2M HILL	Coordinates staffing; directs and oversees project staff; supervises field sampling and coordinates all field activities; ensures onsite compliance with work plan; oversees and ensures safety of onsite personnel.	Mr. Burkingstock has an MS and a BS in Hydrogeology and over 11 years experience; Ms. Struse has an MS in Environmental Engineering, a BS in Chemical Engineering, and over 8 years experience in task management, including staff coordination and supervision, project compliance, and safety assurance

SAP Worksheet #7—Personnel Responsibilities and Qualifications Table (continued)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications ^a
Andrew O'Connor	FTL and SSC	AGVIQ-CH2M HILL	Supervises field sampling and coordinates all field activities; ensures onsite compliance with work plan; oversees and ensures safety of onsite personnel.	BS, Geology; over 8 years experience in well installation and development, soil characterization, groundwater characterization, and environmental remediation
Alicia Nobles	QC Manager	AGVIQ-CH2M HILL	Responsible for daily QC reports, oversight of quality. Monitor and report on subcontractor quality and quantities and audit subcontractors' offsite fabrication. Maintain Submittal Register. Participate in continuous improvement of project team and maintain lessons learned log.	BS, Civil Engineering; over 2 years experience in well installation and development, soil characterization, groundwater characterization, and environmental remediation
Mike Goldman	Health and Safety Officer	AGVIQ-CH2M HILL	Responsible for overall Navy program health and safety performance; reviews project-specific HSP; interacts with SSC to ensure project-specific safety of field personnel.	BS, Biology; over 22 years experience in health and safety, including preparing, implementing, and ensuring compliance with project-specific HSPs
Duane Johnson	Database Manager	Critigen	Uploads validated data to environmental database. Manages sample tracking; coordinates assimilation of data from field collection through analysis, validation, and upload to environmental database; performs data queries for data evaluation and report writing.	BS, Chemistry; over 8 years experience designing data management systems, sample tracking, coordinates e-data deliverables with the laboratory, data validation and quality evaluation, and report preparation
Camden Robinson	Data Validator	AGVIQ-CH2M HILL	Responsible for validating analytical data in accordance with project-specific UFP-SAP.	BA, Chemistry; over 5 years experience in chemistry, including laboratory analysis, sampling, data validation, and field testing
Elizabeth Martin	QAO	GCAL	Responsible for laboratory QA program and review of QC data.	
Elizabeth Martin	Organics Department Manager	GCAL	Responsible for oversight, QC, and data review of organics laboratory.	

SAP Worksheet #7—Personnel Responsibilities and Qualifications Table (continued)

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications^a
Elizabeth Martin	Inorganics Department Manager	GCAL	Responsible for oversight, QC, and data review of inorganics laboratory.	
Elizabeth Martin	PM	GCAL	Laboratory POC and overall manager for analytical work.	
Enid Ortiz Valles	Puerto Rican Chemist	GCAL	Responsible for certifying laboratory data	
TBD	Drilling Subcontractor	Haca & Sierra	Responsible for monitoring well installation.	
TBD	Vegetation Clearance Subcontractor	Aleut Global Solutions	Responsible for vegetation clearance, as necessary, to access sites and sample locations.	
TBD	Surveying Subcontractor	PJDC Professional Land Surveyors	Responsible for horizontal coordinate and vertical elevation surveying of newly installed monitoring wells.	
TBD	Utilities Clearance Subcontractor	One Vision, Inc.	Responsible for locating underground utilities.	
TBD	Remediation Waste Subcontractor	Clean Harbors Caribe Inc.	Responsible for transport and disposal of remediation waste deemed necessary for offsite disposal.	

Notes:

^a Resumes are maintained by the individuals' organizations and are available upon request; upon execution of the project, staff may be removed (if unnecessary to project execution) and other staff may be added or substituted, as necessary and available.

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SAP Worksheet #8—Special Personnel Training Requirements Table

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records/Certificates
Field activities	Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour Training, 8-hour refreshers, as applicable	Various qualified training organizations	Training of AGVIQ-CH2M HILL and subcontractors will be verified as current prior to starting field activities by SSC.	All field personnel	FTLs, field team members, and SSC (AGVIQ-CH2M HILL personnel); drilling subcontractor; remediation waste subcontractor; vegetation clearance subcontractor, excavation subcontractor, geophysical subcontractor, and surveying subcontractor	CH2M HILL Human Resources Department for CH2M HILL personnel; subcontractor organizations for field subcontractors
Field activities	Cardiopulmonary resuscitation (CPR)/First Aid Training	Various qualified training organizations	Training will be verified as current prior to starting field activities.	AGVIQ-CH2M HILL SSC	AGVIQ-CH2M HILL SSC	CH2M HILL Human Resources Department
Field activities	SSC-hazardous waste training	Various qualified training organizations	Training will be verified as current prior to starting field activities by SSC.	AGVIQ-CH2M HILL SSC	AGVIQ-CH2M HILL SSC	CH2M HILL Human Resources Department

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SAP Worksheet #9a—Project Scoping Session Participants Sheet

Project Name: SWMUs 54 and 55					
Projected Date(s) of Sampling: April 2009				Site Name: NAPR	
PM: Tom Beisel				Site Location: Ceiba, Puerto Rico	
Dates of Session: December 16, 2008					
Scoping Session Purpose: Site visit to SWMUs 54 and 55 to familiarize project team with site layout, meet Navy and AGVIQ personnel, and gauge select wells.					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Tom Beisel	Project Manger	AGVIQ-CH2M HILL	(678) 530-4033	Tom.Beisel@ch2m.com	Project Manger
Thomas Kessler	Senior Geologist	AGVIQ-CH2M HILL	(678) 530-4197	Thomas.Kessler@ch2m.com	Senior Geologist
Bryan Burkingstock	Project Geologist	AGVIQ-CH2M HILL	(678) 530-4060	Bryan.Burkingstock@ch2m.com	Task Manager for SWMU 7/8
Kimberley Coke	Project Geologist	AGVIQ-CH2M HILL	(678) 530-4073	Kimberley.Coke@ch2m.com	FTL/SSC
Amanda Struse	Project Engineer	AGVIQ-CH2M HILL	(678) 530-4339	Amanda.Struse@ch2m.com	Task Manager for SWMUs 54 and 55
BT Thomas	Project Geologist	AGVIQ-CH2M HILL	(678) 530-4415	BT.Thomas@ch2m.com	QC Manager
Comments/Decisions: Second visit required to locate wells, clear site, and perform a comprehensive round of groundwater level gauging.					
Action Items: Groundwater level data will be used to determine the direction of groundwater flow and optimize the locations of proposed monitoring and injection wells. Also, determined that clearing of site was necessary to access all proposed new well locations.					
Consensus Decisions: Scheduled trip to perform clearing and gauging. Date set to complete tasks during the week of January 19, 2009.					

SAP Worksheet #9b—Project Scoping Session Participants Sheet

Project Name: SWMUs 54 and 55					
Projected Date(s) of Sampling: April 2009				Site Name: NAPR	
PM: Tom Beisel				Site Location: Ceiba, Puerto Rico	
Dates of Session: January 19 – 23, 2009					
Scoping Session Purpose: Oversight of clearing operations, marking locations of all wells, and comprehensive round of well gauging.					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Thomas Kessler	Senior Geologist	AGVIQ-CH2M HILL	(678) 530-4197	Thomas.Kessler@ch2m.com	Senior Geologist
Bryan Burkingstock	Project Geologist	AGVIQ-CH2M HILL	(678) 530-4060	Bryan.Burkingstock@ch2m.com	Task Manager for SWMU 7/8
Kimberley Coke	Project Geologist	AGVIQ-CH2M HILL	(678) 530-4073	Kimberley.Coke@ch2m.com	FTL/SSC
Philip Jones	Project Engineer	AGVIQ-CH2M HILL	(678) 530-4191	Philip.Jones@ch2m.com	Field Team Personnel
Doug Downey	Senior Engineer	AGVIQ-CH2M HILL	(303) 674-6507	Doug.Downey@ch2m.com	Senior Technology Consultant
Comments/Decisions: Water levels at SWMU 55 were collected. There are not enough existing wells at SWMU 54 to compete a reasonable potentiometric map. Also, it was not possible to locate well 510MW5R at SWMU 54. Locations of proposed wells were staked.					
Action Items: Installation of proposed injection and monitoring wells and some additional clearing.					
Consensus Decisions: Additional clearing is required at SWMU 54 to have sufficient access to proposed well locations. Well 510MW5R must be re-installed.					

SAP Worksheet #9c—Project Scoping Session Participants Sheet

Project Name: SWMUs 54 and 55					
Projected Date(s) of Sampling: April 2009				Site Name: NAPR	
PM: Tom Beisel				Site Location: Ceiba, Puerto Rico	
Dates of Session: February 4, 2009					
Scoping Session Purpose: Present and discuss technical approach for pilot test at SWMUs 54 and 55.					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
David Criswell	BRAC PMO SE RPM	Navy	843-743-2130	david.criswell@navy.mil	Primary Navy POC
Tom Beisel	Project Manger	AGVIQ-CH2M HILL	678-530-4033	Tom.Beisel@ch2m.com	Project manager
Doug Downey	Senior Engineer	AGVIQ-CH2M HILL	303-674-6507	Doug.Downey@ch2m.com	Senior Technology Consultant
Thomas Kessler	Senior Geologist	AGVIQ-CH2M HILL	678-530-4197	Thomas.Kessler@ch2m.com	Senior Geologist
Bryan Burkingstock	Project Geologist	AGVIQ-CH2M HILL	678-530-4060	Bryan.Burkingstock@ch2m.com	Task Manager for SWMU 7/8
Kimberley Coke	Project Geologist	AGVIQ-CH2M HILL	678-530-4073	Kimberley.Coke@ch2m.com	Field Team Leader/Site Safety Coordinator
Theresa Rojas	QA/QC Manager	AGVIQ-CH2M HILL	678-530-4297	Theresa.Rojas@ch2m.com	Program QA/QC Manager
Shruti Shah	Environmental Scientist	AGVIQ-CH2M HILL	678-530-4316	Shruti.Shah@ch2m.com	UFP-SAP Coordinator
Amanda Struse	Project Engineer	AGVIQ-CH2M HILL	678-530-4339	Amanda.Struse@ch2m.com	Task Manager for SWMUs 54 and 55
Comments/Decisions: Technical approach was approved by the Navy RPM.					
Action Items: Complete UFP-SAP with technical approach.					
Consensus Decisions:					

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SAP Worksheet #10—Problem Definition

General

In general, the objective at SWMU 54 is to conduct pilot-scale testing to evaluate the use of in situ biodegradation (ISB) to remediate trichloroethene (TCE) contamination in groundwater and air sparging (AS) to remediate benzene contamination in groundwater. The objective at SWMU 55 is to conduct pilot-scale testing to evaluate the use of in situ chemical oxidation (ISCO) to remediate TCE contamination in groundwater.

Regulatory History

Prior to 1993, environmental activities at the former Naval Station Roosevelt Roads (NSRR), exclusive of underground storage tank (UST) operations, were conducted in compliance with Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) regulations under the Navy's Installation Restoration Program (IRP). On October 20, 1994, the U.S. Environmental Protection Agency (EPA) Region 2 issued a Final RCRA Part B Permit to the NSRR, now NAPR. The permit contained requirements for RCRA facility investigation activities at 24 SWMUs and three areas of concern, including SWMUs 54 and 55. The RCRA Part B Permit, issued for the Defense Reutilization and Marketing Office (DRMO) at NAPR, included provisions for CA under the Hazardous and Solid Waste Amendments (HSWA) provisions of RCRA.

The EPA Region II is the primary agency that regulates environmental activities at the NAPR, and site work is performed under RCRA Administrative Order on Consent-7003.

Conceptual Site Model Summary

This section includes brief discussions on the following topics:

- Physical setting and land use
- Geologic setting
- Hydrology
- Background and potential release history (synopsis of secondary data)
- Contaminant distribution

Physical Setting and Land Use

In 1943, the NSRR was commissioned as a Naval Operations Base and then re-designated as a Naval Station in 1957. The NSRR closed in March of 2004 and the NAPR was established. The NAPR occupies over 8,600 acres at the northeastern most portion of Puerto Rico along the Vieques Passage. The northern entrance to NAPR is about 35 miles east of San Juan, along the coast road (Route 3). The nearest large town is Fajardo, located approximately 10 miles north of NAPR on Route 3. The Town of Ceiba adjoins the west boundary of NAPR (Figures 1 and 2).

SWMU 54

SWMU 54 is the Former Naval Exchange (NEX) Repair/Maintenance Shop (Building 1914), which was constructed in 1979. It is currently unoccupied and lies on approximately 1 acre of land in the Bundy Area of NAPR (Figure 1). A UST was present at the site and used to store fuel until its removal in December 1992 (Blasland, Bouck, and Lee, 1995) (Figure 3). The topography of SWMU 54 consists of a slight slope to the west and a small hill to the east, approximately 100 feet in

elevation. Another small hill lies in the southern portion, which is approximately 50 feet in elevation. The building structure itself consists of a small concrete block building with a center office area and open bays on either side.

SWMU 55

SWMU 55 is located in the eastern portion of NAPR as shown on Figure 2. A substantial structure (Building 46), located on the building pad immediately northeast of 7MW7 (between Forrestal Drive and the well), was destroyed during Hurricane Hugo in September 1989. Building 46 was rebuilt in 1991 as Building 2314; a commercial storage building. Building 2314 was originally a Quonset-style building with a cloth roof. This building was used for cold storage, and partially destroyed during Hurricane Georges in September 1998. Currently, Building 2314 exists as an uncovered concrete foundation with several unused walk-in freezers and buoys placed on top of the foundation.

Geologic Setting

The geology of NAPR consists of the four geologic units: fill, soil consisting of saprolite and partially weathered rock (PWR), bedrock, and marine sediments. The fill material is comprised of fine- to medium-grained sand with varying amounts of silt and clay. The fill occurs at ground surface to depths as great as 25 feet below ground surface (bgs) in areas of the site that have been reworked. The fill is a combination of reworked native soil (silt, clay, and sand) and/or dredge material from Ensenada Honda. Soil consisting of saprolite (clayey-silt to silty-clay with rock fragments) and PWR gabbro bedrock underlies the fill. Soil beneath the fill is comprised of clayey-silt and silty-clay, with rock fragments of varying size. The percentage of rock fragments increases with depth and grades into PWR zone that contains weather gabbro rock fragments with occasional clay seams. There is no well-defined contact between the residual soil and bedrock; rather, a gradational change of decreased weathering and fracturing occurs with increasing depth. The thickness of this soil and PWR unit is variable and in some places is over 40 feet. Bedrock underlies the PWR. Bedrock consists of gabbro that is hard and massive in some places; however, a few zones are highly fractured due to tectonic deformation. The final zone is a zone of marine sediments located in the lowland area of the site near the Ensenada Honda. The sediments consist of silt with lesser amounts of sand and clay with coral and shell fragments. (Baker, 2005)

Hydrology

Groundwater beneath the site occurs at depths ranging from about 4 to 112 feet bgs. The depth to water is greatest in the upland areas of the site and is shallowest nearest the Ensenada Honda. Water level changes in the upland and lowland areas are likely caused by seasonal variations in precipitation. In the lowland area south of Forrestal Drive, water levels fluctuations are also a result of tidal influence. The primary direction of flow is south toward the Ensenada Honda. According to the final CMS, the average hydraulic gradient is 0.0063 foot/foot (Baker, 2005). AGVIQ-CH2M HILL determined an average hydraulic gradient of 0.003 foot/foot at SWMU 54 and 0.007 foot/foot at SWMU 55.

Background and Potential Release History (Synopsis of Secondary Data)

SWMU 54

An UST was present at the site and used to store fuel until its removal in December 1992 (Blasland, Bouck, and Lee, 1995) (Figure 3). The date of installation and type of fuel stored is unknown, but is assumed to be gasoline. The building structure itself consists of a small concrete block building with a center office area and open bays on either side. The building was used to perform

maintenance on vehicles, including oil changes and lubrications. No wastes are known to have been disposed of at the unit and there are no known releases related to the unit (Baker, 2005).

SWMU 55

SWMU 55 is located in the eastern portion of NAPR and is presented on Figure 2. The TCE plume site at Tow Way Fuel Farm (TWFF) is located south of Forrestal Drive near Building 2314 (formerly Building 46). Prior to Hurricane Hugo in 1989, a substantial building existed at this site that was reportedly used for the storage and maintenance of small watercraft. It is unclear to what extent the building was used for storage of materials, such as solvents, and the original source could not be determined (Baker, 2005). However, based on previous environmental investigations conducted at the site, the approximate vertical extent of contamination was estimated to be from 10 to 35 feet bgs, and the aerial extent was estimated to be approximately 150 feet by 180 feet (Baker, 2005).

Contaminant Distribution

SWMU 54

Benzene Plume

According to the final CMS, a benzene plume was identified west of Bairoko Street (Baker, 2005) (Figure 5). The final CMS recommended a pilot-scale test be conducted to evaluate the use of aerobic biodegradation to remediate the benzene plume.

Between August 2009 and April 2010, AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of benzene contamination in groundwater. The investigation confirmed that two benzene plumes above the CAO of 550 micrograms per liter ($\mu\text{g}/\text{L}$) exist: one in a shallow zone from 5 to 15 feet bgs primarily on the west side of Bairoko Street and one in a deep zone from 15 to 25 feet bgs primarily on the east side of Bairoko Street. The investigation also identified a previously unknown area of shallow benzene contamination present in monitoring well 54MW34 located on the southeast side of Bairoko Street near a drainage ditch. During the investigation, the maximum benzene concentration for the shallow zone plume on the west side of Bairoko Street was measured at monitoring well 54MW06 at 14,200 $\mu\text{g}/\text{L}$; the maximum benzene concentration for the shallow zone plume on the southeast side of Bairoko Street was measured at monitoring well 54MW34 at 10,800 $\mu\text{g}/\text{L}$; and the maximum benzene concentrations for the deep zone plume on the east side of Bairoko Street was measured at monitoring well 54MW27 at 7,410 $\mu\text{g}/\text{L}$. (AGVIQ-CH2M HILL, 2010b). Based on the results of the additional investigation, AGVIQ-CH2M HILL recommended installation of additional monitoring wells to determine the extent of benzene in both the shallow and deep zone on the east side of Bairoko Street (AGVIQ-CH2M HILL, 2010b).

AGVIQ-CH2M HILL suspended the ISB pilot-scale test at the benzene plume, and in May 2010, conducted an AS pilot-scale test to evaluate the effectiveness of AS to remediate the benzene plume.

TCE Plume

According to the final CMS, a TCE plume was identified east of Bairoko Street (Baker, 2005) (Figure 4). The final CMS recommended pilot-scale testing to evaluate the use of ISB via enhanced reductive dechlorination (ERD) to remediate the TCE plume.

Between July 2009 and January 2010, AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of TCE contamination in groundwater. A comparison of TCE concentrations reported in the final CMS (Baker, 2005) with the additional investigation concentrations indicated that TCE concentrations have decreased since 2002, and the configuration

of the plume shifted slightly to the south (AGVIQ-CH2M HILL, 2009a). During the investigation, the maximum TCE concentration was measured at injection well 54IW04 at 256 µg/L (AGVIQ-CH2M HILL, 2010a).

Between December 2009 and February 2010, AGVIQ-CH2M HILL conducted an ISB pilot-scale test to evaluate the effectiveness of ISB via ERD to remediate the TCE plume.

SWMU 55

According to the final CMS, environmental investigations conducted at SWMU 55 and the TWFF concluded TCE in groundwater is the only compound in excess of the human health based CAO of 22 µg/L at SWMU 55 (Baker, 2005) (Figure 6). Most recently, a TCE plume delineation and source investigation was performed in September 2003. During this investigation, the maximum TCE concentration in groundwater was measured at monitoring well 7MW7 at 1,800 µg/L (Baker, 2005). Based on the results of this investigation, the final CMS recommended that an ISCO pilot test be performed to evaluate ISCO to address TCE in groundwater at SWMU 55.

Between August 2009 and April 2010, AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of groundwater contamination. During the investigation, TCE was detected in groundwater to a depth of 41 feet bgs, with the greatest TCE concentrations detected in a vertical zone extending from approximately 15 to 25 feet bgs. The maximum TCE concentration was measured at injection well 55IW01 at 33,600 µg/L. A zone exceeding 1,000 µg/L extended from the source area to well pair 7MW23/55MW14. The 1,000 µg/L area, including the source area, has been defined as the target treatment zone for SWMU 55. TCE concentrations decline with groundwater elevation; however, the lateral extent of TCE exceeding the CAO of 22 µg/L increases slightly with depth. (AGVIQ-CH2M HILL, 2010c)

In December 2009, AGVIQ-CH2M HILL conducted an ISCO pilot-scale test to evaluate the effectiveness of ISCO to remediate the TCE plume. The ISCO pilot-scale test indicated limited permanganate persistence, rapid dissipation of permanganate from the source area, and TCE rebound at injection well 55IW01. Based on these results, AGVIQ-CH2M HILL determined that ISCO injections would not be an effective or economical long-term remedy to reduce TCE concentrations in groundwater. Therefore, AGVIQ-CH2M HILL recommended modifying the remedial approach to incorporate excavation to address the northern portion of the source area, completing an additional ISCO application to rapidly reduce TCE mass in the 55IW01 source area, and using ERD to address lingering TCE concentrations in the source area and downgradient plume. (AGVIQ-CH2M HILL, 2010c).

Problem Definition

SWMU 54

Benzene Plume

An additional investigation was performed to delineate the vertical and horizontal extent of benzene exceeding the CAO of 550 µg/L in groundwater at SWMU 54. Based on the investigation, additional monitoring wells were installed to fully delineate the benzene source area plumes.

An AS pilot-scale test was conducted at the benzene side of SWMU 54 to evaluate the radius of influence (ROI) from a single injection well, the effects of groundwater mounding and vapor migration during air injection, the distribution of air in the subsurface at varying injection rates, and the pressure required to achieve adequate distribution of air in the subsurface.

TCE Plume

An additional investigation was performed to delineate the vertical and horizontal extent of TCE exceeding the CAO of 22 µg/L in groundwater at SWMU 54.

An ISB pilot-scale test was conducted at the TCE plume to evaluate the ability to achieve an adequate injection rate, the ability to achieve an adequate ROI, the effectiveness of ISB via ERD to treat TCE in groundwater, the persistence of ISB substrate in the subsurface, the required frequency for re-application, and the impact of site geochemical properties that affect ISB performance.

SWMU 55

An additional groundwater investigation was performed to delineate the vertical and horizontal extent of TCE exceeding the CAO of 22 µg/L in groundwater at SWMU 55. An ISCO pilot-scale test was conducted to evaluate the ability to achieve an adequate injection rate, the ability to achieve an adequate ROI, the effectiveness of ISCO to treat TCE in groundwater, the persistence of sodium permanganate in the subsurface, and the possible rebound of TCE in groundwater.

Based on the groundwater investigation and pilot-scale test, additional monitoring wells were installed to determine if the source area is present upgradient of existing injection well 55IW01.

Environmental Questions to be Answered by the Corrective Measures for SWMUs 54 and 55 (Pilot-Scale Test)

SWMU 54 Benzene Plume

1. Is there an ability to achieve adequate distribution of air in site formation?

Dissolved oxygen (DO) content and oxidation-reduction potential (ORP) of site groundwater will be monitored and recorded during active AS. If DO and ORP increases are not measured at least 15 feet from the AS well, an alternative technology may be considered. The extent of air distribution attainable at a single AS point has significant impact on the cost to complete a full-scale implementation.

2. Is there an ability to achieve adequate ROI at a single sparge well?

The ROI will be measured by monitoring the aquifer response (including water levels and water quality parameters) at monitoring points adjacent to the AS well. If a ROI of 15 feet or greater cannot be achieved during the injections, an alternative technology or an alternative means of introducing material to the subsurface may be considered. The ROI has significant influence on the cost to complete subsurface injections, and a small ROI may make the technology economically infeasible or less implemental than an alternative technology for full-scale application.

3. What is the effectiveness of AS to treat COCs in groundwater?

The potential to effectively treat COCs in groundwater using AS will be based on the distribution of DO on the subsurface during the AS pilot-scale test.

4. What is the extent of contamination in groundwater?

AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of benzene contamination in groundwater. Based on the results of this investigation, AGVIQ-CH2M HILL recommended installation of monitoring wells and collection of water quality samples to complete delineation of the benzene plume at SWMU 54. Water quality samples will be analyzed for benzene using EPA Method 8260B.

SWMU 54 TCE Plume

1. Is there an ability to achieve adequate injection rates in site formation?

Injection rates will be monitored and recorded during active injection. If injection rates of 1 gallon per minute (gpm) or greater cannot be achieved during the injections, an alternative technology or an alternative means of introducing material to the subsurface, such as fracturing of the formation, may be considered. The achievable injection rate has significant influence on the cost to complete subsurface injections and low injection rates may make the technology economically infeasible or less implementable than an alternative technology for full-scale application.

2. Is there an ability to achieve adequate ROI at a single injection point?

The ROI will be measured by monitoring the aquifer response (including water levels and water quality parameters) at injection and monitoring points adjacent to the injection location. If a ROI of 7 to 10 feet or greater cannot be achieved during the injections, an alternative technology or an alternative means of introducing material to the subsurface, such as fracturing of the formation, may be considered. The ROI has significant influence on the cost to complete subsurface injections and a small ROI may make the technology economically infeasible or less implementable than an alternative technology for full-scale application.

3. What is the effectiveness of ISB to treat COCs in groundwater?

The potential to effectively treat COCs in groundwater using ISB will be based on the ability to adequately distribute injectant into the formation. The potential to effectively treat COCs in groundwater within the area affected by pilot-scale testing will be evaluated by 30-day post-injection, 90-day post-injection, and two quarterly groundwater monitoring events following completion of the ISB pilot-scale test.

4. What is the persistence of injectant and required frequency for re-application?

The cost of ISB applications is influenced by the frequency with which the follow-up injections must be conducted (if more than one injection required) to maintain the treatment zone. Substrate persistence will be determined by measuring several groundwater parameters, such as groundwater quality parameters (DO, total organic carbon [TOC], ORP, and conductivity), contaminant concentrations, biodegradation indicators, such as methane, ethane, and ethene (MEE).

5. What is the impact to site geochemical properties, which impact ISB performance?

The efficiency of ISB is dependent on several factors, including site geochemical properties. For example, under certain pH ranges, ISB may be rendered less effective because particular ions or metals needed by the bacteria may not be in solution or the bacteria responsible for ISB may be affected directly (Interstate Technology Regulatory Council [ITRC], 2002).

6. What is the extent of contamination in groundwater?

AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of TCE contamination in groundwater. AGVIQ-CH2M HILL completed delineation of the TCE plume at SWMU 54.

SWMU 55

1. Is there an ability to achieve adequate injection rates in site formation?

Injection rates will be monitored and recorded during active injection. If injection rates of 1 gpm or greater cannot be achieved during the injections, an alternative technology or an alternative means of introducing material to the subsurface, such as fracturing of the formation, may be

considered. The achievable injection rate has significant influence on the cost to complete subsurface injections and low injection rates may make the technology economically infeasible or less implementable than an alternative technology for full-scale application.

2. Is there an ability to achieve adequate ROI at a single injection point?

The ROI will be measured by monitoring the aquifer response (including water levels and water quality parameters) at injection and monitoring points adjacent to the injection location. If a ROI of 7 to 10 feet or greater cannot be achieved during the injections, an alternative technology or an alternative means of introducing material to the subsurface, such as fracturing of the formation, may be considered. The ROI has significant influence on the cost to complete subsurface injections and a small ROI may make the technology economically infeasible or less implemental than an alternative technology for full-scale application.

3. What is the effectiveness of ISCO to treat COCs in groundwater?

The potential to effectively treat COCs in groundwater using ISCO will be based on the ability to adequately distribute injectant into the subsurface. The potential to effectively treat COCs in groundwater within the area affected by pilot-scale testing will be evaluated by three quarterly groundwater monitoring events following completion of the ISCO pilot-scale test.

4. What is the persistence of injectant and required frequency for re-application?

The cost of ISCO applications is influenced by the frequency with which the follow-up injections must be conducted (if more than one injection required) to maintain the treatment zone. Permanganate persistence will be determined by measuring the permanganate and ORP concentration in groundwater.

5. What is the extent of contamination in groundwater?

AGVIQ-CH2M HILL performed an additional investigation to determine the horizontal and vertical extent of TCE contamination in groundwater. Based on the results of this investigation, AGVIQ-CH2M HILL recommended installation of monitoring wells and collection of water quality samples to complete delineation of the TCE plume. Water quality samples will be analyzed for TCE using EPA Method 8260B.

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SAP Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements

1. **Who will use the data and what will the data be used for?**

The Navy, EPA, PREQB, subcontractors, and AGVIQ-CH2M HILL will use the data collected during the additional investigation and pilot-scale tests (as well as relevant historical data) to determine the extent of contamination and effectiveness of pilot-scale testing at SWMUs 54 and 55.

2. **What are the Project Action Limits (PALs)?**

Pilot testing will be conducted to evaluate ISCO and ISB for treatment of contaminants in groundwater and to develop design parameters for full-scale CA. If the pilot tests are successful and these technologies are implemented at SWMUs 54 and 55, then the CAOs will be addressed. The CAOs for SWMU 54 are 22 µg/L for TCE and 550 µg/L for benzene. The CAO for SWMU 55 is 22 µg/L for TCE.

3. **What types of data are needed (matrix, target analytes, analytical groups, field screening, onsite analytical or offsite laboratory techniques, sampling techniques?)**

Worksheets #10 and 17 contain detailed information on the types of data needed for this project. Worksheet #12 defines the matrices, analytical groups, and target analytes for each site.

No field screening will be conducted during the pilot testing activities. Sample analyses for COCs at both sites will be conducted by an offsite laboratory in accordance with Worksheets #15, 19, 23, 24, 25, 28, and 30. Analyses for sodium permanganate (NaMnO₄) will be conducted onsite using a colorimeter.

Drilling, monitoring well installation and developing, groundwater sampling, and related activities will be done in accordance with the applicable standard operating procedures (SOPs) in Appendix B.

4. **How “good” do the data need to be in order to support the environmental decision?**

All data will be validated against QA/QC criteria and measurement performance criteria listed in this SAP and certified by a Puerto Rican chemist. Level IV package and QC sampling are required. Data will be used to evaluate the effectiveness of the pilot testing at SWMUs 54 and 55. QC data requirements are detailed in Worksheet #20.

Data need to be sufficient to meet the following:

Visual Observations – Visual observations will be used at various sites to determine presence or absence of NaMnO₄ and hydrocarbon staining, and soil moisture conditions. As such, the data are considered qualitative in that they do not need to provide an exact or quantified value.

Onsite Analytical Data – At SWMU 55, a colorimeter will be used to determine the concentration of NaMnO₄ in groundwater following the completion of injection activities. Colorimeter results will be used to determine distribution of NaMnO₄ in the subsurface. Therefore, the onsite analytical results need to be adequate to determine if the NaMnO₄ concentrations are above 2 milligrams per liter (mg/L).

Offsite Analytical Data – The data need to be “good” enough to evaluate results of pilot testing at SWMUs 54 and 55. Ensuring data are “good” enough for this purpose is done via employing appropriate analytical protocol, validating the resulting data, including QA/QC samples to verify proper sampling and analysis protocol, and performing a data quality evaluation (DQE) to assess the availability and usability of the data for the intended purpose. Each of these is further discussed below:

- Appropriate Analytical Protocol – See Worksheets #15, 19, 23, 24, 25, 28, and 30, and Item 5 below.
- Data Validation – Validation of data increases the level of confidence in a data set for a particular data use. The particular type and level of validation necessary to achieve acceptable confidence is subjective. In other words, the appropriate type and level of data validation is not an absolute. Rather, it is data use- and data user-specific. For the groundwater sampling events, analyses for potential contaminants will be certified by a Puerto Rican chemist, 90 percent of the data will be validated by a AGVIQ-CH2M HILL data validator, and 10 percent of the data will be validated by a third party validator using guidance from the validation criteria outlined by the U.S. Department of Defense (DOD) Quality System Manual (QSM). The validation criteria and guidance documents are listed in Worksheet #36. These documents will help the validators create a thorough and systematic approach to the validation process. The data validator will also recalculate 100 percent of the results from the raw laboratory data, which may identify laboratory errors in identification or quantification, if present.
- QA/QC Samples – During the pilot test, QA/QC samples will be collected as a check on sampling and analytical protocol. Like data validation, the appropriate type and quantity of QA/QC samples is not an absolute. For this pilot test, field duplicates will be collected at a frequency of 1 per 10 field samples per matrix. Field duplicates help assess sample collection techniques and laboratory precision. Matrix spike/matrix spike duplicates (MS/MSDs) are collected at a frequency of 1 pair per 20 field samples per matrix. The frequency is such that there is one MS/MSD pair per laboratory analytical batch. MS/MSD samples are often required by the analytical method and/or data validation guidance. Equipment blanks are collected at a frequency of 1 per day per decontaminated equipment. Equipment blanks help assess equipment decontamination techniques and identify when contamination may have been carrying over from one sample location to another. It is important to maintain this equipment blank frequency because the equipment blank is collected after visiting the most contaminated location, and it is important to not associate too many locations with the potentially-contaminated equipment blank. Trip blanks are collected at a frequency of 1 per cooler containing volatiles. Trip blanks accompany the empty sample containers while they are stored at the laboratory or shipped to the site, and while they are full and shipped back to the laboratory. Trip blanks are useful for assessing whether or not there is any contamination during periods of time when the samples are not directly supervised. No field blanks will be collected unless on a particular day of sampling, the ambient conditions suggest airborne particulates may contaminate the samples being collected.
- Data Quality Evaluation – In order to support the environmental decision, each result must be *available* to and *usable* for the project team. All data sets will undergo a DQE prior to using the data to make site-specific determinations. The terms *data availability* and *data usability* and the DQE process in general are described in Worksheet #37.

5. **How much data should be collected (number of samples for each analytical group, matrix, and concentration)?**

SWMU 54 TCE Plume

Between August 2009 and January 2010, AGVIQ-CH2M HILL performed baseline site characterization sampling in two events: Phase I and Phase II.

Phase I: In August 2009, AGVIQ-CH2M HILL performed Phase I baseline site characterization sampling to verify the current concentrations of TCE in groundwater and verify the TCE plume delineation as presented in the final CMS (Baker, 2005). A total of 18 groundwater samples were collected from 9 new monitoring wells (510MW5R and 54MW07 through 54MW14) (see Figure 4) and analyzed for TCE, cis-1,2-dichloroethene (DCE), vinyl chloride, iron, manganese, sulfate, sulfide, TOC, DHE, MEE, and alkalinity (AGVIQ-CH2M HILL, 2009a). In addition, groundwater samples were collected from wells 510MW5R and 54MW07 through 54MW14 and analyzed for dissolved iron and dissolved manganese. Based on the results of this event, AGVIQ-CH2M HILL recommended installation of four new monitoring wells to delineate the horizontal extent of the TCE plume (AGVIQ-CH2MHILL, 2009a).

Phase II: Between December 2009 and January 2010, AGVIQ-CH2M HILL performed Phase II baseline site characterization sampling to complete delineation of the TCE plume. A total of nine groundwater samples were collected from four new monitoring wells (54MW15 through 54MW18) installed around the perimeter of the TCE plume and five new injection wells (54IW01 through 54IW05) (see Figure 4) and analyzed for TCE, DCE, vinyl chloride, iron, manganese, sulfate, sulfide, TOC, MEE, and alkalinity (AGVIQ-CH2M HILL, 2010a).

ISB Pilot-Scale Test: Between December 2009 and February 2010, AGVIQ-CH2M HILL conducted an ISB pilot-scale test.

Post-Injection Performance Monitoring: In February and April 2010, AGVIQ-CH2M HILL conducted 30-day and 90-day post-injection performance monitoring events. A total of 13 groundwater samples were collected from monitoring wells 510MW5R and 54MW07 through 54MW18 and analyzed for TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity.

Field parameters, including DO, ORP, pH, temperature, specific conductance, and turbidity, were measured during purging with a YSI multiprobe. The number of QA/QC samples collected is detailed in Worksheet #20. All groundwater sampling was conducted according to the SOPs presented in Appendix B.

Both aqueous and soil waste from the benzene and TCE plume at SWMU 54 were containerized together. Two aqueous waste characterization samples were collected and analyzed for RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH. Three soil waste characterization samples were collected and analyzed for Toxicity Characteristic Leaching Procedure (TCLP) compounds (see Worksheet #15 for specific compounds).

Quarterly Performance Monitoring: Beginning 150 days post-injection, four quarterly performance monitoring events will be conducted. A total of 13 groundwater samples will be collected from monitoring wells 510MW5R and 54MW07 through 54MW18 and analyzed for

TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity.

SWMU 54 Benzene Plume

In August 2009, AGVIQ-CH2M HILL collected four soil samples during installation of two monitoring wells (54MW02 and 54MW03) (see Figure 5). and analyzed for benzene. Two soil samples were collected for each boring; one in the interval exhibiting the highest headspace reading and one in the interval exhibiting staining and/or a hydrocarbon odor.

Between August 2009 and April 2010, AGVIQ-CH2M HILL performed baseline site characterization in three events: Phase I, Phase II, and Phase III.

Phase I: In August 2009, AGVIQ-CH2M HILL performed Phase I baseline site characterization sampling to verify the current concentrations of benzene in groundwater and verify the benzene plume delineation presented in the final CMS (Baker, 2005). A total of six groundwater samples were collected from six new monitoring wells (54MW01 through 54MW06) (see Figure 5) and analyzed for alkalinity, TOC, nitrate, sulfide, chemical oxygen demand (COD), dissolved iron, benzene, and sulfate (AGVIQ-CH2MHILL, 2009a). Based on the results of this event, AGVIQ-CH2M HILL recommended delaying the ISB pilot-scale test on the benzene plume, collecting and analyzing groundwater samples for volatile organic compounds (VOCs) from previously installed monitoring wells 510DW1, 510DW2, and 510MW1 through 510MW4, and installing five monitoring well pairs and analyzing for VOCs to delineate the extent of the benzene plume (AGVIQ-CH2M HILL, 2009a).

Phase II: Between December 2009 and January 2010, AGVIQ-CH2M HILL performed Phase II baseline site characterization sampling to further characterize the benzene plume. A total of 22 groundwater samples were collected from 10 new monitoring wells (54MW19 through 54MW28) and 12 previously installed monitoring wells (510DW1, 510DW2, 510MW1 through 510MW4, and 54MW01 through 54MW06) (see Figure 5) and analyzed for VOCs (AGVIQ-CH2MHILL, 2010a). Based on the results of this event, AGVIQ-CH2M HILL recommended installing 13 new monitoring wells and analyzing for benzene only to further characterize the benzene plume (AGVIQ-CH2M HILL, 2010a).

Phase III: In April 2010, AGVIQ-CH2M HILL performed Phase III baseline site characterization sampling to further characterize the benzene plume. A total of 13 groundwater samples were collected from 13 new monitoring wells (54MW29 through 54MW41) (see Figure 5) and analyzed for benzene only, with the exception of wells 54MW34 through 54MW36, which were also analyzed for TCE (AGVIQ-CH2MHILL, 2010b). Based on the results of this event, AGVIQ-CH2M HILL recommended installing three new monitoring wells and analyzing for benzene only to further characterize the benzene plume (AGVIQ-CH2M HILL, 2010b). AGVIQ-CH2M HILL will perform Phase IV baseline site characterization sampling, including installing three new monitoring wells and collecting and analyzing three groundwater samples for benzene, to complete characterization of the benzene plume.

AS Pilot-Scale Test: In May 2010, AGVIQ-CH2M HILL conducted an AS pilot-scale test. During the active pilot-scale test, VOCs were periodically measured at both the wellheads of select monitoring points and at known underground utility locations using a photoionization detector. In addition, field parameters, including DO, ORP, and conductivity, were measured at select monitoring points.

Field parameters, including DO, ORP, pH, temperature, specific conductance, and turbidity, were measured during purging with a YSI multimeter. The number of QA/QC samples is detailed in Worksheet #20. All groundwater sampling was conducted according to the SOPs presented in Appendix B.

Both aqueous and soil waste from the benzene and TCE plume at SWMU 54 were containerized together. Two aqueous waste characterization samples were collected and analyzed for RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH. Three soil waste characterization samples were collected and analyzed for TCLP compounds (see Worksheet #15 for specific compounds).

At the conclusion of monitoring well installation at the benzene plume, one aqueous waste characterization sample will be collected and analyzed for RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH. One soil waste characterization sample will be collected and analyzed for TCLP compounds (see Worksheet #15 for specific compounds).

SWMU 55 TCE Plume

Between July 2009 and April 2010, AGVIQ-CH2M HILL performed baseline site characterization sampling in four events: Phase I through Phase IV.

Phase I: In July 2009, AGVIQ-CH2M HILL performed Phase I baseline site characterization sampling to verify results of the last sampling event conducted in 2003 (Baker, 2005), evaluate the current extent of groundwater contamination, ensure the location of the TCE plume has not shifted since 2003, and possibly refine the pilot-scale test injection well locations. A total of six groundwater samples were collected from six existing monitoring wells (7MW7, 7MW10, and 7MW21 through 7MW24) (see Figure 6) and analyzed for TCE. Based on the results of this event, AGVIQ-CH2M HILL recommended delaying the ISCO pilot-scale test and installing five new monitoring well pairs to delineate the vertical and horizontal extent of the TCE plume (AGVIQ-CH2M HILL, 2009b).

Total Oxidant Demand (TOD) Test: A TOD test is a bench-scale test used to evaluate the total oxidant demand resulting from organic material (both contaminants and naturally occurring matter) in soil and groundwater. Because most oxidant demand results from naturally occurring organic material in the subsurface, the site-specific TOD is a significant factor in determining permanganate dosing rates and potential persistence of permanganate in the subsurface.

The TOD test was conducted using potassium permanganate (KMnO_4). Because the permanganate ion is the oxidizing agent of interest, it may be used in the lab or the field as either NaMnO_4 or KMnO_4 . The bench-scale test consisted of 12 reaction vessels, 3 for each soil sample. The reaction vessels were each comprised of 50 grams of dry soil and 100 milliliters of KMnO_4 solution. Each soil sample was dosed with three KMnO_4 concentrations (one per reaction vessel), and the KMnO_4 concentration was then measured over time. Because the resulting TOD (and subsurface permanganate persistence) is dependent on the initial permanganate concentration, three KMnO_4 concentrations (500 mg/L, 5,000 mg/L, and 10,000 mg/L) were tested. The permanganate doses were chosen to mimic field conditions as the injected permanganate solution radiates from the injection point and becomes more dilute with distance and time. The TOD testing was conducted for 2 weeks, and the TOD was

calculated for each reaction vessel by determining the decline in permanganate mass per mass soil treated over time.

Phase II: In November 2009, AGVIQ-CH2M HILL performed Phase II baseline site characterization sampling to delineate the vertical and horizontal extent of the TCE plume. A total of 14 groundwater samples were collected from five new well pairs (55MW01 through 55MW10) and four new injection wells (55IW01 through 55IW04) (see Figure 6) and analyzed for TCE. Based on the results of this event, AGVIQ-CH2M HILL recommended installing 10 new monitoring wells to further characterize the horizontal and vertical extent of the TCE plume (AGVIQ-CH2M HILL, 2009c).

Phase III: In February 2010, AGVIQ-CH2M HILL performed Phase III baseline site characterization sampling to further characterize the horizontal and vertical extent of the TCE plume. A total of nine groundwater samples were collected from nine new monitoring wells (55MW11, 55MW12, and 55MW14 through 55MW20) (see Figure 6) and analyzed for TCE. Proposed monitoring well 55MW13, downgradient of the ISCO injection pilot-scale test area, was not installed due to the proximity of the pilot-scale test area (AGVIQ-CH2M HILL, 2009c). Based on the results of this event, AGVIQ-CH2M HILL recommended installing four new monitoring wells to further characterize the horizontal and vertical extent of the TCE plume (AGVIQ-CH2M HILL, 2010d).

Phase IV: In April 2010, AGVIQ-CH2M HILL performed Phase IV baseline site characterization sampling to further characterize the horizontal and vertical extent of the TCE plume. A total of four groundwater samples were collected from four new monitoring wells (55MW13 and 55MW21 through 55MW23) (see Figure 6) and analyzed for TCE (AGVIQ-CH2M HILL, 2010c).

ISCO Pilot-Scale Test: In December 2009, AGVIQ-CH2M HILL conducted an ISCO pilot-scale test.

Quarterly Performance Monitoring: In January 2010, AGVIQ-CH2M HILL performed the first quarterly performance monitoring event following the ISCO pilot-scale test. A total of 20 groundwater samples were collected from 16 monitoring wells (55MW01 through 55MW10, 7MW07, 7MW10, and 7MW21 through 7MW24) and four injection wells (55IW01 through 55IW04) (see Figure 6) and analyzed for TCE. NaMnO_4 was present in five of the groundwater samples from wells 7MW07 and 55IW01 through 55IW04. These groundwater samples were not submitted for chemical analysis and NaMnO_4 was measured with a field colorimeter. Due to a rapid reaction between TCE and permanganate, it is assumed that no significant TCE concentration will exist in the presence of permanganate.

In April 2010, AGVIQ-CH2M HILL performed the second quarterly monitoring event. A total of 23 groundwater samples were collected from 19 monitoring wells (55MW01 through 55MW12, 55MW14, 7MW07, 7MW10, and 7MW21 through 7MW24) and four injection wells (55IW01 through 55IW04) (see Figure 6) and analyzed for TCE.

Field parameters, including DO, turbidity, conductivity, pH, temperature, and ORP, were recorded during well purging with a YSI or similar field instrument. All groundwater sampling was conducted according to the SOPs presented in Appendix B.

Three aqueous waste characterization samples were collected and analyzed for RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide),

ignitability, and corrosivity as pH. One soil waste characterization sample was collected and analyzed for TCLP compounds (see Worksheet #15 for specific compounds).

The third quarterly performance monitoring event will include the collection of 23 groundwater samples from 19 monitoring wells (55MW01 through 55MW12, 55MW14, 7MW07, 7MW10, and 7MW21 through 7MW24) and four injection wells (55IW01 through 55IW04) (see Figure 6) and analysis for TCE. Field parameters, including DO, turbidity, conductivity, pH, temperature, and ORP, will be recorded during well purging with a YSI or similar field instrument. Groundwater sampling will be conducted according to the SOPs presented in Appendix B.

Phase V: Based on the results of the quarterly performance monitoring and Phase IV baseline sampling events, AGVIQ-CH2M HILL recommended installation of two monitoring wells upgradient of the ISCO pilot-scale test injection area to define the source area (AGVIQ-CH2M HILL, 2010c). AGVIQ-CH2M HILL will perform Phase V baseline site characterization sampling, including installing two new monitoring wells (55MW24 and 55MW25) and collecting and analyzing two groundwater samples for TCE, to complete characterization of the TCE plume.

At the conclusion of monitoring well installation, one aqueous waste characterization sample will be collected and analyzed for RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH. One soil waste characterization sample will be collected and analyzed for TCLP compounds (see Worksheet #15 for specific compounds).

6. Where, when, and how should the data be collected/generated?

Figures 4 and 5 show the sampling locations for SWMU 54, Figure 6 shows the sampling locations for SWMU 55. The SOPs for groundwater sampling and NaMnO₄ analyses are provided in Appendix B, and the sampling schedule is outlined in Worksheet #16. Sample collection will be conducted according to Table 1.

TABLE 1
 Sample Collection Schedule

Sampling Activity	Location	Timeframe	Groundwater Sampling Parameters
Baseline Sampling Event	SWMUs 54 and 55	Conducted prior to injection work and as required for additional investigation	<ul style="list-style-type: none"> • Water levels • Select VOCs • Select monitored natural attenuation (MNA) parameters • Groundwater quality parameters
Injection Performance Monitoring	SWMU 54	Conducted during active injection	<ul style="list-style-type: none"> • Water levels • Groundwater quality parameters
Injection Performance Monitoring	SWMU 55	Conducted during active injection	<ul style="list-style-type: none"> • Water levels • NaMnO₄

TABLE 1
 Sample Collection Schedule

Sampling Activity	Location	Timeframe	Groundwater Sampling Parameters
Post-Injection Performance Monitoring	SWMU 54	Conducted 30 days, 90 days, and quarterly for four quarters after injection is complete	<ul style="list-style-type: none"> • Water levels • Select VOCs • Select MNA parameters • Groundwater quality parameters
Post-Injection Performance Monitoring	SWMU 55	Conducted quarterly for three quarters after injection is complete	<ul style="list-style-type: none"> • Water levels • NaMnO₄ • Groundwater quality parameters • Select VOCs

7. Who will collect and generate the data? How will the data be reported?

AGVIQ-CH2M HILL will collect the data samples. Laboratory analytical data will be generated by GCAL and field data will be generated by AGVIQ-CH2M HILL. The data will be evaluated and reported in the SWMU 54 and SWMU 55 annual reports.

8. How will the data be archived?

The electronic data will be loaded into the Navy Installation Restoration Information System (NIRIS) database. Raw data, as well as data summary tables, will be included in the annual reports. Hardcopy data will be released to the Navy following completion of the project. All field data will be collected on the appropriate field forms or logbook. This information will be placed with the project files.

9. List the project quality objectives in the form of if/then qualitative and quantitative statements.

SWMU 54 TCE Plume

ISB has been widely used to address VOCs in groundwater, including TCE and benzene. According to the final CMS, enhanced natural attenuation was recommended for the TCE plume at SWMU 54; however, the time required to achieve CAOs using MNA was not acceptable (Baker, 2005). Therefore, ISB was selected to enhance the natural processes already occurring and reduce the time required to achieve CAOs. The remediation method for the TCE plume east of Bairoko Street will be ISB via ERD. This technology will be implemented through the injection of organic substrates, such as a mixture of food-grade vegetable oil and lactate. This is a proven method of accelerating the biodegradation of groundwater contaminants. Data obtained from the pilot-scale test, along with the performance monitoring results, will be summarized in the SWMU 54 annual report.

The primary study question for this phase of work is as follows: Is ISB a viable technology for full scale application?

Planned actions include the following:

- If the pilot-scale testing is considered successful, then ISB will be considered for full-scale application for the TCE plume at SWMU 54.
- If the pilot-scale testing results in adequate contaminant treatment, full-scale CA may not be required.
- If the pilot-scale testing is not considered successful, then an alternate remedial technology will be evaluated.

SWMU 54 Benzene Plume

According to the final CMS, enhanced natural attenuation was recommended for the benzene plume at SWMU 54; however, the time required to achieve CAOs using MNA was not acceptable (Baker, 2005). Therefore, ISB was selected to enhance the natural processes already occurring and reduce the time required to achieve CAOs (AGVIQ-CH2M HILL, 2009d). However, baseline characterization results confirmed significantly greater benzene concentrations in groundwater than those presented in the final CMS (Baker, 2005). ISB of the benzene plume is not considered a viable option due to the presence of significantly higher benzene concentrations. Therefore, AS was selected for evaluation.

AS is an in situ remedial technology that involves the injection of air into the saturated zone approximately 10 to 15 feet below the water table to volatilize VOCs and to promote aerobic biodegradation of hydrocarbon. AS is a proven remedial technology with relatively low capital and operational costs, and the technology is consistent with the approved method of using in situ methods to promote aerobic degradation of the benzene plume. Additionally benzene has a low vapor pressure and is easily volatilized by aeration.

The primary study question for this phase of work is as follows: Is AS a viable technology for full-scale application?

Planned actions include:

- If the pilot-scale testing is considered successful, then AS will be considered for full-scale application for the benzene plume at SWMU 54.
- If the pilot-scale testing results in adequate contaminant treatment, full-scale CA may not be required.
- If the pilot-scale testing is not considered successful, then an alternate remedial technology will be evaluated.

SWMU 55

Chemical oxidation is an aggressive technology that may be used in situ to rapidly treat a variety of organic contaminants in groundwater. ISCO is typically employed for the degradation of chlorinated ethenes, such as TCE (Siegrist et al., 2001), and is most effective in source zone treatment. Generally, ISCO is implemented through the subsurface injection of chemical oxidants, resulting in contaminants being oxidized to carbon dioxide and other innocuous compounds. The most widely used oxidants include hydrogen peroxide (Fenton's reagent), permanganate (MnO_4^-) as either NaMnO_4 or as KMnO_4 , persulfate, and ozone. Permanganate was selected for the pilot test at SWMU 55 due to the stability of the MnO_4^- ion in

the subsurface resulting in long persistence in groundwater and subsequent long-term treatment of dissolved TCE. In addition, MnO_4^- does not require activation and therefore is more easily implemented than persulfate or potentially peroxide.

Based on the results of the TCE plume delineation and source investigation, the final CMS recommended that an ISCO pilot test be performed to evaluate ISCO to address TCE in groundwater at SMWU 55 (Baker, 2005). Data results of the KMnO_4 pilot test will be reported in the SWMU 55 annual report.

The primary study question for this phase of work is as follows: Is ISCO a viable technology for full-scale application?

Planned actions include:

- If the pilot-scale testing is considered successful, then ISCO will be considered for full-scale application at SWMU 55.
- If the pilot-scale testing results in adequate contaminant treatment, full-scale CA may not be required.
- If the pilot-scale testing is not considered successful, then an alternate remedial technology will be evaluated.

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
MS/MSD	VOCs – SW-846 8260B Water/GCAL/SOP-GCMSV-003	One MS/MSD per 20 samples	Accuracy/Bias/Precision	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL; with the exception of common field/laboratory contaminants	S
Cooler Temperature Indicator		One per cooler	Accuracy/Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
MS/MSD	Dissolved metals – Iron and Manganese SW-846 6010B Water/ GCAL/SOP-MET-010	One MS/MSD per 20 samples	Accuracy/Bias/Precision	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample (continued)

QC Sample	Analytical Group	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment/Rinsate Blanks	Dissolved metals – Iron and Manganese SW-846 6010B Water/ GCAL/SOP-MET-010	One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL; with the exception of common field/laboratory contaminants	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
MS/MSD	Sulfate and Nitrate EPA 300.0 Water/ GCAL/SOP-WL-042	One MS/MSD per 20 samples	Accuracy/Bias/Precision	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample (continued)

QC Sample	Analytical Group	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
MS	Sulfide SM 4500-S D Water/ GCAL/SOP-WL-033	One MS per 20 samples	Accuracy/Bias	MS Recovery 75-125%	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
MS	Total Organic Carbon SM 5310B Water/ GCAL/SOP-WL043	One MS per 20 samples	Accuracy/Bias	MS Recovery 75-125%	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample (continued)

QC Sample	Analytical Group	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Data Completeness Check	Total Organic Carbon	NA	Data Completeness	100%	S&A
Comparability Check	SM 5310B Water/ GCAL/SOP-WL043	As required per sampling event	Comparability	NA	NA
MS/MSD	Dissolved MEE RSK-175 Water/ GCAL/SOP-GC-024	One MS/MSD per 20 samples	Accuracy/Bias/Precision	MS/MSD Recovery 30-170% Relative percent difference (RPD) less than or equal to 20%	A
Field Duplicates		One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL; with the exception of common field/laboratory contaminants	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
MS		COD HACH 8000 Water/ GCAL/SOP-WL-021	One MS per 20 samples	Accuracy/Bias	MS Recovery 75-125%
Field Duplicates	One field duplicate per 10 samples		Precision	NA	S&A
Equipment/Rinsate Blanks	One equipment blank per day per decontaminated equipment		Bias/Contamination	No target analytes greater than or equal to the RL	S

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample (continued)

QC Sample	Analytical Group	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Cooler Temperature Indicator	COD HACH 8000 Water/ GCAL/SOP-WL-021	One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
Field Duplicates	Alkalinity SM 2320B Water/ GCAL/SOP-WL-063	One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks		One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA
MS/MSD		VOCs – Benzene only SW-846 8260B Soil/ GCAL/SOP-GCMSV-003	One MS/MSD per 20 samples	Accuracy/Bias/Precision	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3

SAP Worksheet #12—Measurement Performance Criteria Table – Field QC Sample (continued)

QC Sample	Analytical Group	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicates	VOCs – Benzene only	One field duplicate per 10 samples	Precision	NA	S&A
Equipment/Rinsate Blanks	SW-846 8260B Soil/ GCAL/SOP-GCMSV-003	One equipment blank per day per decontaminated equipment	Bias/Contamination	No target analytes greater than or equal to the RL; with the exception of common field/laboratory contaminants	S
Cooler Temperature Indicator		One per cooler	Accuracy/ Representativeness	Samples should be received at the lab at less than or equal to 6°C	S
Data Completeness Check		NA	Data Completeness	100%	S&A
Comparability Check		As required per sampling event	Comparability	NA	NA

SAP Worksheet #13—Secondary Data Criteria and Limitations Table

The table below provides general information on how secondary data will be used and the limitations on their use. Following the general table below, secondary data criteria and limitations tables are presented for each site where historical analytical data exist (applicable to the scope of work covered by this SAP), specifically to address the use and limitations of the historical analytical data.

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Site background information	Prepared by: Baker Environmental, Inc. Report Title: Final Corrective Measures Study Final Report for SWMUs 54 and 55 Date: August 29, 2005	Site histories for SWMUs 54 and 55	Data will be used to provide site history summaries and to gain an understanding of historical activities that led to a potentially CERCLA-related release.	Site investigations may not have full linear or vertical extent. Also, most recent groundwater data is from 2002.
Site background information	Prepared by: Baker Environmental, Inc. Report Title: Final Corrective RCRA Facility Investigation Report for SWMUs 53 and 54, Naval Station Roosevelt Roads, Ceiba, Puerto Rico Date: July 2003	Site history for SWMU 54	Data will be used to provide a site history summary and to gain an understanding of historical activities that led to a potentially CERCLA-related release.	Limited data set.

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SAP Worksheet #14—Summary of Project Tasks

General Protocol

Mobilization

Mobilization will include AGVIQ-CH2M HILL staff and subcontractors traveling to the island. Mobilization will occur only after the following tasks have been completed:

- The SAP has been approved by the stakeholders
- Utility clearance

Utility Clearance

Drilling subcontractor will coordinate the utility clearance on potential subsurface utilities at all locations prior to the start of field work.

SWMU 54 TCE Plume

Installation of Monitoring Wells

A total of 13 monitoring wells (510MW5R and 54MW07 through 54MW18) were installed, as shown on Figure 4. Well placement rationale for wells 54MW07 through 54MW14 is presented in the SWMU 54 work plan (AGVIQ-CH2M HILL, 2009d), and rationale for wells 54MW15 through 54MW18 is presented in a technical memorandum (AGVIQ, CH2M HILL, 2009a). In addition to the eight wells proposed in the work plan, well 510MW5R was installed to replace historical well location 510MW5 (AGVIQ, CH2M HILL, 2009d).

The wells were installed using hollow stem auger (HSA) drilling techniques. The well installation was performed in accordance with the SOP presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials also conformed to the requirements of the Puerto Rico Standards and Regulations as required by the PREQB.

Installation of Injection Wells

Prior to initiating the pilot-scale test, five injection wells (54IW01 through 54IW05) were installed in the vicinity of 510MW5R, as shown on Figure 4. The injection well locations were revised from those presented in the work plan; the rationale for this revision is presented in a technical memorandum (AGVIQ, CH2M HILL, 2009a).

The wells were installed using HSA drilling techniques. The well installation was performed in accordance with the SOP presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials also conformed to the requirements of the Puerto Rico Standards and Regulations as required by PREQB.

Preliminary Injection Test

Preliminary injection tests were originally planned to determine the ability to inject fluids in the subsurface prior to procuring and shipping chemical for the pilot-scale test injections (AGVIQ, CH2M HILL, 2009d). However, during installation of site wells, groundwater recovery rates indicated a geologic lithology with lower than expected hydraulic conductivity. Therefore, slug tests were conducted, instead of the preliminary injection tests, to evaluate the aquifer conductivity.

Substrate Injection

The TCE pilot-scale test involved the injection of a 2.5 (weight) percent emulsified vegetable oil (EVO) at five locations within the SWMU 54 source area. Initially, injection was conducted only at injection well 54IW01, while monitoring wells 510MW5R and 54MW09 through 54MW14 were used as monitoring points. During injection at 54IW01, groundwater samples were collected periodically (minimum of once per day) from the monitoring points and evaluated visually for cloudiness. The following groundwater quality parameters were recorded during purging of the select monitoring points: DO, ORP, specific conductivity, pH, and turbidity. The data were used to evaluate a potential ROI and the achievable injection rate. In addition, groundwater levels were measured twice per day. After sufficient data were attained to evaluate the ROI, injection commenced at two to four of the injection wells concurrently. Up to 4,700 gallons of the 2.5 percent EVO solution were injected at each injection well. This equates to approximately 1,040 pounds of EVO injected at each well.

The EVO injection was conducted using a mobile system equipped with one mixing tank, a centrifugal pump, and a generator. Instrumentation for monitoring the system, such as flow meters and pressure gauges, was also included. The EVO injection solution was mixed onsite using a batch mixing process. The injection work was conducted in accordance with the HSP and SAP.

Post-Injection Performance Monitoring

Sampling was conducted 30 and 90 days after the EVO injection event. Sampling activities will be continued quarterly for 1 year after completion of the injection event. See Worksheet #11 for total number of samples and specific analysis.

SWMU 54 Benzene Plume

Installation of Monitoring Wells

A total of 29 monitoring wells (54MW01 through 54MW06, 54MW19 through 54MW28, and 54MW29 through 54MW41) were installed, as shown on Figure 5. Placement rationale for wells 54MW01 through 54MW06 is presented in the work plan (AGVIQ, CH2M HILL, 2009d); rationale for wells 54MW19 through 54MW28 is presented in a technical memorandum (AGVIQ, CH2M HILL, 2009a); and rationale for wells 54MW29 through 54MW41 is presented in a Phase II technical memorandum (AGVIQ, CH2M HILL, 2010a). In addition, a total of three monitoring wells (54MW42 through 54MW44) will be installed, as shown on Figure 5. The rationale for these wells is presented in a Phase III technical memorandum (AGVIQ, CH2M HILL, 2010b).

The wells are installed using HSA drilling techniques. The well installation is performed in accordance with the SOP presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials conform to the requirements of the Puerto Rico Standards and Regulations as required by PREQB.

Installation of Air Sparge Well

One AS well (54AS01) was installed, as shown on Figure 5. The location of the AS well was selected based on its location in the benzene plume, as well as the proximity and spacing of adjacent monitoring wells to be utilized for monitoring. The well network surrounding the proposed location of the pilot-scale test AS well will provide sufficient information to determine technology effectiveness and provide design parameters for full-scale implementation.

The well was installed using HSA drilling techniques. The well installation was performed in accordance with the SOPs presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials also conformed to the requirements of the Puerto Rico Standards and Regulations as required by PREQB.

Air Sparging Pilot-Scale Test

The AS pilot-scale test consisted of air injection at 54AS01 at varying flow rates and pressures, while monitoring wells 510DW1, 510DW2, 510MW1, 54MW01, 54MW03, 54MW06, 54MW19, 54MW20, 54MW21, 54MW27, 54MW28, 54MW32, 54MW33, 54MW36, 54MW37, 54MW38, and 54MW41 were used as monitoring points. Groundwater levels were measured prior to initiation of and after completion of the pilot-scale test. VOCs were periodically measured at both the wellheads of the monitoring points and at known underground utility locations using a photoionization detector. In addition, YSI data loggers were placed in select monitoring points to monitor the water level and the following groundwater quality parameters: DO, ORP, and conductivity.

SWMU 55

Installation of Monitoring Wells

A total of 21 monitoring wells (55MW01 through 55MW23) were installed, as shown on Figure 6. Placement rationale for wells 55MW01 through 55MW10 is presented in a technical memorandum (AGVIQ, CH2M HILL, 2009b); rationale for wells 55MW11, 55MW12, 55MW14, and 55MW20 is presented in a Phase II technical memorandum (AGVIQ, CH2M HILL, 2009c); and rationale for wells 55MW13, 55MW21, 55MW22, and 55MW23 is presented in a Phase III technical memorandum (AGVIQ, CH2M HILL, 2010d). In addition, a total of two monitoring wells (55MW24 and 55MW25) will be installed, as shown on Figure 6. The rationale for these wells is presented in a Phase IV technical memorandum (AGVIQ, CH2M HILL, 2010c).

The wells are installed using HSA drilling techniques, with the exception of wells where HSA drilling techniques are used to advance borings to auger refusal; at these wells, air rotary drilling techniques are used to complete the boring to the proposed depth. The well installation is performed in accordance with the SOPs presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials conform to the requirements of the Puerto Rico Standards and Regulations as required by the PREQB.

Installation of Injection Wells

Prior to initiating the pilot-scale test, four injection wells (55IW01 through 55IW04) were installed in the source area. The injection well locations were revised from those presented in the work plan (AGVIQ-CH2M HILL, 2009e); the rationale for this revision is presented in a technical memorandum (AGVIQ, CH2M HILL, 2009b).

The wells were installed using HSA drilling techniques. The well installation was performed in accordance with the SOP presented in Appendix B and the general requirements of the *EPA Region 4 2008 Field Branches Quality System and Technical Procedures* (EPA, 2009). Well installation procedures and materials also conformed to the requirements of the Puerto Rico Standards and Regulations as required by the PREQB.

Preliminary Injection Test

Preliminary injection tests were originally planned to be conducted to evaluate the ability to physically inject a liquid solution into the formation (AGVIQ-CH2M HILL, 2009e). However, due to the high levels of TCE (greater than 14,000 µg/L) encountered in the pilot-scale test injection area, the preliminary injection test was not conducted to avoid unintentional dispersion of the TCE plume without concurrent treatment. In the place of the preliminary injection test, falling and rising head slug tests were completed to characterize the aquifer conductivity (AGVIQ-CH2M HILL, 2009b).

TOD Testing

TOD tests were conducted on soils collected from four vertical intervals during installation of injection well 55IW01: 18 to 22 feet bgs and 22 to 26 feet bgs, and 55IW04: 25 to 29 feet bgs and 35 to 41 feet bgs. All samples were collected from the decomposed gabbro since no lithified gabbro was encountered within 40 feet bgs. These vertical intervals were also representative of the entire pilot-scale test injection interval of 15 to 40 feet bgs. Worksheet #11 presents the TOD sampling process. The SWMU 55 work plan discusses the rationale for TOD testing (AGVIQ-CH2M).

ISCO Injection

The ISCO pilot-scale test originally planned for the injection of a 30- to 50-gram-per-liter (g/L) KMnO_4 solution at four locations within the SWMU 55 source area (AGVIQ-CH2M HILL, 2009e); however, based on the TOD test results (refer to Worksheet #11), a 16.5 g/L NaMnO_4 solution (1.5 percent solution) was injected. Initially, injection was conducted only at injection well 55IW02, while monitoring wells 7MW07, 7MW22, 7MW23 and 7MW24 and injection wells 55IW01, 55IW03, and 55IW04 were used as monitoring points. During injection at 55IW02, groundwater samples were collected periodically (minimum of twice per day) from the monitoring points and analyzed for NaMnO_4 using a field colorimeter. The data were used to evaluate a potential ROI and the achievable injection rate. In addition, groundwater levels were measured twice per day. Up to 5,000 gallons of the 1.5 percent NaMnO_4 solution were injected at each injection well. This equates to approximately 622 pounds of NaMnO_4 injected at each well.

The NaMnO_4 injection was conducted using a mobile system equipped with one mixing tank, a centrifugal pump, and a generator. Instrumentation for monitoring the system, such as flow meters and pressure gauges, was also included. The NaMnO_4 injection solution was mixed onsite using a batch mixing process. The SWMU 55 work plan presents the ISCO injection process (AGVIQ-CH2M).

Post-Injection Performance Monitoring

Beginning 30 days after completion of the injection event, two quarterly performance monitoring events were conducted. A third performance monitoring event will be conducted. See Worksheet #11 for total number of samples and specific analysis.

Sampling Locations and Quantities

SWMU 54

Groundwater

During the baseline site characterization, a total of 27 groundwater samples were collected at the SWMU 54 TCE plume and 41 groundwater samples were collected at the SWMU 54 benzene plume. After installation of three additional monitoring wells (54MW42 through 54MW44) is complete, three additional groundwater samples will be collected. During the installation of

monitoring wells 54MW02 and 54MW03, four soil samples were collected for chemical analysis. All monitoring and injection well locations are illustrated on Figure 3.

The samples are labeled based on the monitoring or injection well location ID. In addition to the horizontal coordinate information, the task order and sampling date is added to the sample nomenclature. For example, for a groundwater sample collected at injection well 55IW01, the sample identification (ID) would be labeled JM04-55IW01-mmddyy, and for a soil sample collected at monitoring well 55MW01, the sample ID would be labeled JM04-55MW01(sampling interval)-mmddyy.

SWMU 55

During the baseline site characterization at SWMU 55, a total of 33 groundwater samples were collected. After installation of two additional monitoring wells (55MW24 and 55MW25) is complete, two additional groundwater samples will be collected. During installation of injection wells 55IW01 and 55IW04, two soil samples were collected for use in the TOD test. All monitoring and injection well locations are illustrated on Figure 6.

The samples are labeled based on the monitoring or injection well location ID. In addition to the horizontal coordinate information, the task order and sampling date is added to the sample nomenclature. For example, for a groundwater sample collected at injection well 55IW01, the sample ID would be labeled JM04-55IW01-mmddyy, and for a soil sample collected at monitoring well 55MW01, the sample ID would be labeled JM04-55MW01(sampling interval)-mmddyy.

Sampling Procedures

Groundwater

All groundwater sampling procedures are presented in Appendix B.

Soil

SWMU 54

Two soil samples were collected and analyzed for benzene from wells 54MW02 and 54MW03. As the boring was advanced, soil samples were collected every 6 inches and screened using a flame ionization detector (FID). Following collection, the sample was split for headspace screening, lithologic description, and chemical analysis. One portion of the sample was preserved as the possible chemical analysis sample by immediately placing the soil in the appropriate sample vessel. Another portion of the sample was immediately placed in a plastic bag, sealed, and allowed to equilibrate for 10 minutes. The bag was then be pierced with the FID probe and a headspace reading was recorded. The headspace reading was used to evaluate the relative concentration of contaminants and to aid in sample selection for laboratory analysis. The soil sample having the highest headspace reading within each boring was submitted for analysis. In addition, one soil sample from each boring was submitted for analysis due to staining and/or a hydrocarbon odor. Soil samples were analyzed for benzene.

SWMU 55

During installation of injection wells 55IW01 and 55IW04, soil samples were collected for use in the TOD test. All samples were collected from the decomposed gabbro since no lithified gabbro was encountered within 40 feet bgs. In total, approximately 2 kilograms of each soil type (approximately four 200-milliliter soil sample jars) were collected, stored under ice, and shipped to the TOD test laboratory.

Waste Characterization

The composite aqueous waste characterization samples are collected in the following manner:

1. Collect a bailer full of aqueous sample from both the top and bottom of each 55-gallon drum or poly tank containing aqueous waste generated during the well installation or purging activities and decant the water into a glass container to form a composite aqueous sample.
2. Gently stir the sample in the container and pour the composite water sample into the appropriate sample jars.
3. Close the jars and label and package the sample for shipment to the laboratory for chemical analysis.

The composite soil waste characterization samples are collected in the following manner:

1. Collect a spoonful of soil from the top of each 55-gallon drum or roll-off container containing soil waste generated during the installation of monitoring or injection wells and place in a bowl. Using a hand auger, collect a spoonful of soil from the bottom of each container and place in a bowl. Mix the soil to form a composite soil sample.
2. Gently stir the sample in the container and place the composite soil sample into the appropriate sample jars.
3. Close the jars and label and package the sample for shipment to the laboratory for chemical analysis.

Sampling Equipment

The following equipment is used during the sampling work:

- Nitrile or latex gloves
- Durable ice chest (20-gallon) for shipping samples
- Ice for preserving samples
- Laboratory supplied sampling containers (jars/bottles)
- 55-gallon drums (U.S. Department of Transportation-approved), roll-off containers, and poly tanks for containerizing waste
- Peristaltic pump, tubing, and filters to sample monitoring wells
- YSI meter
- LDO meter
- Colorimeter

Sampling Container, Analytical Methods, Preservatives, and Holding Time

The analytical method, preservative, sampling containers, and holding time for the soil samples are presented in Worksheet #15. Worksheet #19 presents all of the analytical SOP requirements, including TCLP and RCRA compounds (see Worksheet #15 for specific compounds). The sample containers are provided by a NELAP-certified and Navy-approved laboratory. This laboratory is

responsible for the chemical analysis of the groundwater, soil, and waste samples and QA/QC samples.

Equipment Decontamination

An area has been designated for the decontamination of equipment and the storage of all waste. All decontaminated materials will be handled only with new, unused nitrile or latex gloves to avoid contamination. Decontamination SOPs are presented in Appendix B.

Sample Packaging, Marking, Labeling, Shipping, and Chain-of-Custody

Samples must be preserved to ensure that the samples are received at the laboratory at a temperature less than or equal to 4 degrees Celsius (°C). Ice will be placed on top of sample containers and the ice chest will be wrapped with duct tape. Custody seals will be applied upon completing the inspection of the ice chest integrity and appropriate documentation.

All samples must be identified using a standard tag attached to the sample container and will include the following information (Appendix C):

- Project name
- Project number
- Sample ID
- Date and time of sample collection
- Sampler name(s)
- Analyses
- Preservatives

Chain-of-custody records are used to record the custody of all samples. An example of the chain-of-custody is included as Appendix C. The following information must be supplied in the indicated spaces:

- Client name
- Project number
- Project name and site location
- Sample identification number of all samples included in the shipment
- Sample description
- Date and time of sample collection
- Name, company, and signature of field technician collecting samples
- Name and signature of laboratory custodian receiving the samples

Quality Control

All QC samples are listed in Worksheet #20. In reference to the field tasks, all field work will be overseen by an FTL who is responsible for the QC of the sampling to make sure the proper work plans are followed for each task.

Accuracy and Precision

The QA/QC field program will be continuously implemented during all sampling activities. Prior to beginning any sampling activities, a decontamination area will be designed for cleaning both sampling and storage tools and containers. Upon completion of sampling, samples will be labeled and placed in an ice chest. All of the sampling information will be recorded in the field book and in

the chain-of-custody. Decontamination procedures are discussed below and will be followed before moving to another sample location.

Improper sample handling may alter the accuracy of the analytical results. Consequently, the samples will only be collected by persons wearing disposable nitrile or latex gloves. At each sampling point, the sampling personnel will wear a new pairs of gloves. QA/QC will consist of obtaining equipment blanks, MS/MSDs and field duplicates. Field duplicate samples will be collected at a frequency of 1 per every 10 samples. Equipment blanks will be collected at a frequency of one per day per decontaminated equipment. MS/MSD will be collected at a frequency of 1 per every 20 samples.

Equipment Blanks

One equipment blank per day per decontaminated equipment will be prepared in the field. A jar containing de-ionized water will be opened in the field and poured over or through cleaned sampling equipment before equipment blank sample collection. The flushing water will be collected in a 1-liter amber bottle and sent to the laboratory for analysis. The equipment blank will constitute a test of effectiveness of sampling equipment decontamination.

Field Duplicates

One sample per 10 soil samples will be split into two separate portions. These two portions will be collected at the same time as two separate samples and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of both samples give a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedure.

Storage

All analytical data will be stored on CH2M HILL's SQL Server Data Warehouse, after which the finalized data will be uploaded to the NIRIS database as part of AVGIQ-CH2M HILL final delivery package. Project records will be recorded in annual reports, and this information will be available as public information. Project files will be stored for 7 years at the following location:

Iron Mountain/Safesite
660 Distribution Road
Atlanta, GA 30336

Sample Analysis

The laboratory will maintain, test, inspect, and calibrate analytical instruments (Worksheets #24 and 25). The laboratory will process and prepare samples for analysis. The laboratory will analyze soil and groundwater samples for various groups of parameters as shown in Worksheets #15 and 18.

Data Management

The Project Database Manager, Duane Johnson, a subcontractor with Critigen, is responsible for data tracking and storage. In addition, a third party data validator will receive 10 percent of all analytical data from the laboratory, and the remaining 90 percent will be validated internally by the AVGIQ-CH2M HILL data validator prior to its use by the Navy.

Procedures for Recording and Correcting Data

All field data will be recorded in field logbooks and updated in the electronic Field Input Sheets to be uploaded in the Database or recorded electronically via the Mobile Integrated Sample Tracking Personal Digital Assistant for upload in the Database.

Project Assessment/Audit: Worksheets #31 and 32.

Data Validation: Worksheets #35 and 36.

Data Usability Assessment: Worksheet #37.

Remediation Waste Management

Remediation waste will be managed and disposed of in accordance with the Waste Management Plan in the work plans for SWMUs 54 and 55.

Environmental Protection Plan

General controls implemented during remediation-related construction activities to prevent pollution and protect the environment are presented in the work plans for SWMUs 54 and 55.

Quality Control Plan

Quality administrators and the sampling inspections associated with SWMUs 54 and 55 are presented in the work plans for SWMUs 54 and 55.

Surveying

The coordinate locations and elevations of the installed wells will be determined by a land surveyor registered in Puerto Rico. The wells will be surveyed relative to a previously established benchmark. The horizontal location will be surveyed to an accuracy of 0.1 foot, and the ground surface and top of casing elevations will be surveyed to an accuracy of 0.01 foot.

Baseline Site Characterization and Pilot-Scale Test Report

Results of the baseline site characterization and the pilot-scale tests will be reported in the Corrective Measures Study Addendums for SWMUs 54 and 55 (forthcoming).

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SAP Worksheet #15—Reference Limits and Evaluation Table

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
Matrix: Groundwater						
Analytical Group: VOCs, Metals, MNA Parameters						
Acetone	67-64-1	NA	NA	NA	25 µg/L	0.791 µg/L
Benzene	71-43-2	550 µg/L	Final CMS	550 µg/L	5 µg/L	0.0747 µg/L
Bromodichloromethane	75-27-4	NA	NA	NA	5 µg/L	0.0574 µg/L
Bromoform	75-25-2	NA	NA	NA	5 µg/L	0.198 µg/L
Bromomethane	74-83-9	NA	NA	NA	5 µg/L	0.148 µg/L
2-Butanone	78-93-3	NA	NA	NA	5 µg/L	0.405 µg/L
Carbon Disulfide	75-15-0	NA	NA	NA	5 µg/L	0.179 µg/L
Carbon Tetrachloride	56-23-5	NA	NA	NA	5 µg/L	0.0825 µg/L
Chlorobenzene	108-90-7	NA	NA	NA	5 µg/L	0.119 µg/L
Chloroethane	75-00-3	NA	NA	NA	5 µg/L	0.140 µg/L
Chloroform	67-66-3	NA	NA	NA	5 µg/L	0.287 µg/L
Chloromethane	74-87-3	NA	NA	NA	5 µg/L	0.134 µg/L
Cyclohexane	110-82-7	NA	NA	NA	5 µg/L	0.0722 µg/L
Dibromochloromethane	124-48-1	NA	NA	NA	5 µg/L	0.036 µg/L
1,2-Dibromo-3-chloropropane	96-12-8	NA	NA	NA	5 µg/L	0.172 µg/L
1,2-Dibromoethane	106-93-4	NA	NA	NA	5 µg/L	0.0651 µg/L
1,3-Dichlorobenzene	541-73-1	NA	NA	NA	5 µg/L	0.0937 µg/L
1,4-Dichlorobenzene	106-46-7	NA	NA	NA	5 µg/L	0.129 µg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
1,2-Dichlorobenzene	95-50-1	NA	NA	NA	5 µg/L	0.102 µg/L
Dichlorodifluoromethane	75-71-8	NA	NA	NA	5 µg/L	0.0608 µg/L
1,1-DCA	75-34-3	NA	NA	NA	5 µg/L	0.065 µg/L
1,2-DCA	107-06-2	NA	NA	NA	5 µg/L	0.083 µg/L
1,1-DCE	75-35-4	NA	NA	NA	5 µg/L	0.119 µg/L
Cis-1,2-DCE	156-59-2	NA	NA	NA	5 µg/L	0.103 µg/L
trans-1,2-DCE	156-60-5	NA	NA	NA	5 µg/L	0.0955 µg/L
1,2-Dichloropropane	78-87-5	NA	NA	NA	5 µg/L	0.0559 µg/L
cis-1,3-Dichloropropene	10061-01-5	NA	NA	NA	5 µg/L	0.040 µg/L
trans-1,3-Dichloropropene	10061-02-6	NA	NA	NA	5 µg/L	0.0561 µg/L
Ethylbenzene	100-41-4	NA	NA	NA	5 µg/L	0.0522 µg/L
2-Hexanone	591-78-6	NA	NA	NA	5 µg/L	0.101 µg/L
Methyl Acetate	79-20-9	NA	NA	NA	5 µg/L	0.373 µg/L
Methylene Chloride	75-09-2	NA	NA	NA	10 µg/L	0.142 µg/L
Methylcyclohexane	108-87-2	NA	NA	NA	5 µg/L	0.0456 µg/L
4-Methyl-2-pentanone	108-10-1	NA	NA	NA	5 µg/L	0.531 µg/L
Methyl tert-Butyl Ether	1634-04-4	NA	NA	NA	5 µg/L	0.099 µg/L
Styrene	100-42-5	NA	NA	NA	5 µg/L	0.0453 µg/L
1,1,2,2-Tetrachloroethane	79-34-5	NA	NA	NA	5 µg/L	0.105 µg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
Tetrachloroethene	127-18-4	NA	NA	NA	5 µg/L	0.0998 µg/L
Toluene	108-88-3	NA	NA	NA	5 µg/L	0.082 µg/L
1,2,4-Trichlorobenzene	120-82-1	NA	NA	NA	5 µg/L	0.107 µg/L
1,1,1-TCA	71-55-6	NA	NA	NA	5 µg/L	0.055 µg/L
1,1,2-TCA	79-00-5	NA	NA	NA	5 µg/L	0.093 µg/L
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA	NA	NA	5 µg/L	0.103 µg/L
Trichloroethene	79-01-6	22 µg/L	Final CMS	22 µg/L	5 µg/L	0.1182 µg/L
Trichlorofluoromethane	75-69-4	NA	NA	NA	5 µg/L	0.072 µg/L
Vinyl chloride	75-01-4	NA	NA	NA	5 µg/L	0.1552 µg/L
Xylenes, total	1330-20-7	NA	NA	NA	10 µg/L	0.334 µg/L
Iron (Dissolved)	7439-89-6	NA	NA	NA	100 µg/L	22 µg/L
Manganese (Dissolved)	7439-96-5	NA	NA	NA	15 µg/L	0.2 µg/L
Sulfate	14808-79-8	NA	NA	NA	500 µg/L	80 µg/L
Nitrate	14797-55-8	NA	NA	NA	500 µg/L	20 µg/L
Sulfide	18496-25-8	NA	NA	NA	20 µg/L	5.3 µg/L
TOC	NA	NA	NA	NA	1000 µg/L	149 µg/L
Dissolved Methane	74-82-8	NA	NA	NA	5 µg/L	0.1163 µg/L
Dissolved Ethane	74-84-0	NA	NA	NA	1 µg/L	0.0241 µg/L
Dissolved Ethene	74-85-1	NA	NA	NA	1 µg/L	0.0298 µg/L
Alkalinity	NA	NA	NA	NA	1000 µg/L	367 µg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
COD	NA	NA	NA	NA	10 mg/L	2 mg/L
Matrix: Groundwater						
Analytical Group: Microbiology						
Dehalococcoides Ethenogenes	NA	NA	NA	NA	500 cells/sample	100 cells/sample
Matrix: Soil						
Analytical Group: VOCs						
Benzene	71-43-2	NA	NA	NA	5 µg/Kg	0.2163 µg/Kg
Matrix: Soil Waste Characterization						
Analytical Group: TCLP Compounds						
Benzene	71-43-2	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.2 mg/L	0.000260 mg/L
Butanone, 2- (Methyl ethyl ketone)	78-93-3	200 mg/L	40 CFR 261.30 Table 1	200 mg/L	0.2 mg/L	0.00705 mg/L
Carbon Tetrachloride	56-23-5	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.2 mg/L	0.00624 mg/L
Chlorobenzene	108-90-7	100 mg/L	40 CFR 261.30 Table 1	100 mg/L	0.2 mg/L	0.0110 mg/L
Chloroform (Trichloromethane)	67-66-3	6 mg/L	40 CFR 261.30 Table 1	6 mg/L	0.2 mg/L	0.00658 mg/L
1,4-Dichlorobenzene	106-46-7	7.5 mg/L	40 CFR 261.30 Table 1	7.5 mg/L	0.05 mg/L	0.0011 mg/L
Dichloroethane, 1,2 (1,2-DCA)	107-06-2	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.2 mg/L	0.00359 mg/L
1,1-Dichloroethene	75-35-4	0.7 mg/L	40 CFR 261.30 Table 1	0.7 mg/L	0.2 mg/L	0.00802 mg/L
Tetrachloroethene	127-18-4	0.7 mg/L	40 CFR 261.30 Table 1	0.7 mg/L	0.2 mg/L	0.00613 mg/L
TCE	79-01-6	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.2 mg/L	0.00473 mg/L
Vinyl Chloride	75-01-4	0.2 mg/L	40 CFR 261.30 Table 1	0.2 mg/L	0.2 mg/L	0.00621 mg/L
Cresol	108-39-4	200 mg/L	40 CFR 261.30 Table 1	200 mg/L	0.1 mg/L	0.0021 mg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
2,4-Dinitrotoluene	121-14-2	0.1 mg/L	40 CFR 261.30 Table 1	0.1 mg/L	0.05 mg/L	0.0012 mg/L
Hexachlorobenzene	118-74-1	0.1 mg/L	40 CFR 261.30 Table 1	0.1 mg/L	0.05 mg/L	0.0014 mg/L
Hexachlorobutadiene	87-68-3	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.05 mg/L	0.0013 mg/L
Hexachloroethane	67-72-1	3 mg/L	40 CFR 261.30 Table 1	3 mg/L	0.05 mg/L	0.0047 mg/L
Nitrobenzene	98-95-3	2 mg/L	40 CFR 261.30 Table 1	2 mg/L	0.05 mg/L	0.0007 mg/L
Pentachlorophenol	87-86-5	100 mg/L	40 CFR 261.30 Table 1	100 mg/L	0.25 mg/L	0.0138 mg/L
Pyridine	110-86-1	5 mg/L	40 CFR 261.30 Table 1	5 mg/L	0.05 mg/L	0.0017 mg/L
2,4,5-Trichlorophenol	95-95-4	400 mg/L	40 CFR 261.30 Table 1	400 mg/L	0.05 mg/L	0.0017 mg/L
2,4,6-Trichlorophenol	88-06-2	2 mg/L	40 CFR 261.30 Table 1	2 mg/L	0.05 mg/L	0.0016 mg/L
Chlordane, technical	57-74-9	0.03 mg/L	40 CFR 261.30 Table 1	0.03 mg/L	0.025 mg/L	0.00016 mg/L
Endrin	72-20-8	0.02 mg/L	40 CFR 261.30 Table 1	0.02 mg/L	0.001 mg/L	0.00003 mg/L
HCH, gamma- (BHC, gamma-) (Lindane)	58-89-9	0.4 mg/L	40 CFR 261.30 Table 1	0.4 mg/L	0.0005 mg/L	0.00003 mg/L
Heptachlor	76-44-8	0.008 mg/L	40 CFR 261.30 Table 1	0.008 mg/L	0.0005 mg/L	0.00003 mg/L
Heptachlor epoxide	1024-57-3	0.008 mg/L	40 CFR 261.30 Table 1	0.008 mg/L	0.0005 mg/L	0.00004 mg/L
Methoxychlor	72-43-5	10 mg/L	40 CFR 261.30 Table 1	10 mg/L	0.025 mg/L	0.00005 mg/L
Toxaphene	8001-35-2	0.5 mg/L	40 CFR 261.30 Table 1	0.5 mg/L	0.25 mg/L	0.00050 mg/L
2,4-Dichlorophenoxyacetic acid	94-75-7	10 mg/L	40 CFR 261.30 Table 1	10 mg/L	0.005 mg/L	0.00005 mg/L
2,4,5-TP (Silvex)	93-72-1	1 mg/L	40 CFR 261.30 Table 1	1 mg/L	0.005 mg/L	0.00006 mg/L
Arsenic	7440-38-2	5 mg/L	40 CFR 261.30 Table 1	5 mg/L	0.2 mg/L	0.0038 mg/L
Barium	7440-39-3	100 mg/L	40 CFR 261.30 Table 1	100 mg/L	1.0 mg/L	0.00052 mg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
Cadmium	7440-43-9	1 mg/L	40 CFR 261.30 Table 1	1 mg/L	0.01 mg/L	0.00017 mg/L
Chromium (total)	7440-47-3	5 mg/L	40 CFR 261.30 Table 1	5 mg/L	0.05 mg/L	0.00030 mg/L
Lead	7439-92-1	5 mg/L	40 CFR 261.30 Table 1	5 mg/L	0.1 mg/L	0.0027 mg/L
Mercury	7439-97-6	0.2 mg/L	40 CFR 261.30 Table 1	0.2 mg/L	0.002 mg/L	0.000066 mg/L
Selenium	7782492	1 mg/L	40 CFR 261.30 Table 1	1 mg/L	0.1 mg/L	0.0045 mg/L
Silver	7440224	5 mg/L	40 CFR 261.30 Table 1	5 mg/L	0.05 mg/L	0.00062 mg/L
Aroclor-1016	12674-11-2	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00020 mg/L
Aroclor-1221	11104-28-2	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00024 mg/L
Aroclor-1232	11141-16-5	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00038 mg/L
Aroclor-1242	53469-21-9	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00040 mg/L
Aroclor-1248	12672-29-6	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00026 mg/L
Aroclor-1254	11097-69-1	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00040 mg/L
Aroclor-1260	11096-82-5	50 mg/L	40 CFR 761.2	50 mg/L	0.01 mg/L	0.00009 mg/L
Matrix: Liquid Waste Characterization						
Analytical Group: RCRA Compounds						
Benzene	71-43-2	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.2 mg/L	0.000260 mg/L
Butanone, 2- (Methyl ethyl ketone)	78-93-3	200 mg/L	40 CFR 261.24 Table 1	200 mg/L	0.2 mg/L	0.00705 mg/L
Carbon Tetrachloride	56-23-5	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.2 mg/L	0.00624 mg/L
Chlorobenzene	108-90-7	100 mg/L	40 CFR 261.24 Table 1	100 mg/L	0.2 mg/L	0.0110 mg/L
Chloroform (Trichloromethane)	67-66-3	6 mg/L	40 CFR 261.24 Table 1	6 mg/L	0.2 mg/L	0.00658 mg/L
1,4-Dichlorobenzene	106-46-7	7.5 mg/L	40 CFR 261.24 Table 1	7.5 mg/L	0.05 mg/L	0.0011 mg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
Dichloroethane, 1,2 (1,2-DCA)	107-06-2	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.2 mg/L	0.00359 mg/L
1,1-Dichloroethene	75-35-4	0.7 mg/L	40 CFR 261.24 Table 1	0.7 mg/L	0.2 mg/L	0.00802 mg/L
Tetrachloroethene	127-18-4	0.7 mg/L	40 CFR 261.24 Table 1	0.7 mg/L	0.2 mg/L	0.00613 mg/L
TCE	79-01-6	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.2 mg/L	0.00473 mg/L
Vinyl Chloride	75-01-4	0.2 mg/L	40 CFR 261.24 Table 1	0.2 mg/L	0.2 mg/L	0.00621 mg/L
Cresol	108-39-4	200 mg/L	40 CFR 261.24 Table 1	200 mg/L	0.1 mg/L	0.0021 mg/L
2,4-Dinitrotoluene	121-14-2	0.1 mg/L	40 CFR 261.24 Table 1	0.1 mg/L	0.05 mg/L	0.0012 mg/L
Hexachlorobenzene	118-74-1	0.1 mg/L	40 CFR 261.24 Table 1	0.1 mg/L	0.05 mg/L	0.0014 mg/L
Hexachlorobutadiene	87-68-3	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.05 mg/L	0.0013 mg/L
Hexachloroethane	67-72-1	3 mg/L	40 CFR 261.24 Table 1	3 mg/L	0.05 mg/L	0.0047 mg/L
Nitrobenzene	98-95-3	2 mg/L	40 CFR 261.24 Table 1	2 mg/L	0.05 mg/L	0.0007 mg/L
Pentachlorophenol	87-86-5	100 mg/L	40 CFR 261.24 Table 1	100 mg/L	0.25 mg/L	0.0138 mg/L
Pyridine	110-86-1	5 mg/L	40 CFR 261.24 Table 1	5 mg/L	0.05 mg/L	0.0017 mg/L
2,4,5-Trichlorophenol	95-95-4	400 mg/L	40 CFR 261.24 Table 1	400 mg/L	0.05 mg/L	0.0017 mg/L
2,4,6-Trichlorophenol	88-06-2	2 mg/L	40 CFR 261.24 Table 1	2 mg/L	0.05 mg/L	0.0016 mg/L
Chlordane, technical	57-74-9	0.03 mg/L	40 CFR 261.24 Table 1	0.03 mg/L	0.025 mg/L	0.00016 mg/L
Endrin	72-20-8	0.02 mg/L	40 CFR 261.24 Table 1	0.02 mg/L	0.001 mg/L	0.00003 mg/L
HCH, gamma- (BHC, gamma-) (Lindane)	58-89-9	0.4 mg/L	40 CFR 261.24 Table 1	0.4 mg/L	0.0005 mg/L	0.00003 mg/L
Heptachlor	76-44-8	0.008 mg/L	40 CFR 261.24 Table 1	0.008 mg/L	0.0005 mg/L	0.00003 mg/L
Heptachlor epoxide	1024-57-3	0.008 mg/L	40 CFR 261.24 Table 1	0.008 mg/L	0.0005 mg/L	0.00004 mg/L

SAP Worksheet #15—Reference Limits and Evaluation Table (continued)

Analyte	CAS Number	Project Action Limit	Project Action Limit Reference	Project Quantitation Limit Goal	GCAL	
					Quantitation Limit	Method Detection Limit
Methoxychlor	72-43-5	10 mg/L	40 CFR 261.24 Table 1	10 mg/L	0.025 mg/L	0.00005 mg/L
Toxaphene	8001-35-2	0.5 mg/L	40 CFR 261.24 Table 1	0.5 mg/L	0.25 mg/L	0.00050 mg/L
2,4-Dichlorophenoxyacetic acid	94-75-7	10 mg/L	40 CFR 261.24 Table 1	10 mg/L	0.005 mg/L	0.00005 mg/L
2,4,5-TP (Silvex)	93-72-1	1 mg/L	40 CFR 261.24 Table 1	1 mg/L	0.005 mg/L	0.00006 mg/L
Arsenic	7440-38-2	5 mg/L	40 CFR 261.24 Table 1	5 mg/L	0.2 mg/L	0.0038 mg/L
Barium	7440-39-3	100 mg/L	40 CFR 261.24 Table 1	100 mg/L	1.0 mg/L	0.00052 mg/L
Cadmium	7440-43-9	1 mg/L	40 CFR 261.24 Table 1	1 mg/L	0.01 mg/L	0.00017 mg/L
Chromium (total)	7440-47-3	5 mg/L	40 CFR 261.24 Table 1	5 mg/L	0.05 mg/L	0.00030 mg/L
Lead	7439-92-1	5 mg/L	40 CFR 261.24 Table 1	5 mg/L	0.1 mg/L	0.0027 mg/L
Mercury	7439-97-6	0.2 mg/L	40 CFR 261.24 Table 1	0.2 mg/L	0.002 mg/L	0.000066 mg/L
Selenium	7782492	1 mg/L	40 CFR 261.24 Table 1	1 mg/L	0.1 mg/L	0.0045 mg/L
Silver	7440224	5 mg/L	40 CFR 261.24 Table 1	5 mg/L	0.05 mg/L	0.00062 mg/L
Aroclor-1016	12674-11-2	NA	NA	NA	0.01 mg/L	0.00020 mg/L
Aroclor-1221	11104-28-2	NA	NA	NA	0.01 mg/L	0.00024 mg/L
Aroclor-1232	11141-16-5	NA	NA	NA	0.01 mg/L	0.00038 mg/L
Aroclor-1242	53469-21-9	NA	NA	NA	0.01 mg/L	0.00040 mg/L
Aroclor-1248	12672-29-6	NA	NA	NA	0.01 mg/L	0.00026 mg/L
Aroclor-1254	11097-69-1	NA	NA	NA	0.01 mg/L	0.00040 mg/L
Aroclor-1260	11096-82-5	NA	NA	NA	0.01 mg/L	0.00009 mg/L

SAP Worksheet #16—Project Schedule

Sampling Event	Start Date	End Date
SWMU 54 Benzene		
Installation of 6 Monitoring Wells	August 2009	August 2009
Phase I Baseline Characterization Event	September 2009	September 2009
Aquifer Slug Testing	October 2009	October 2009
Installation of 10 Monitoring Wells	November 2009	December 2009
Phase II Baseline Characterization Event	December 2009	January 2010
Installation of 13 Monitoring Wells	February 2010	March 2010
Phase III Baseline Characterization Event and Installation of 1 AS Well	April 2010	April 2010
AS pilot-Scale Test	May 2010	May 2010
Phase IV Baseline Characterization Event	October 2010	October 2010
SWMU 54 TCE		
Installation of 9 Monitoring Wells	July 2009	July 2009
Phase I Baseline Characterization Event	August 2009	August 2009
Aquifer Slug Testing and Installation of 1 Injection Well	October 2009	October 2009
Installation of 4 Monitoring Wells	November 2009	November 2009
Phase II Baseline Characterization Event	December 2009	January 2010
ISB Pilot-Scale Test	December 2009	February 2010
30-Day Post-Injection Performance Monitoring	February 2010	February 2010
90-Day Post-Injection Performance Monitoring	April 2010	April 2010
1 st Quarter Post-Injection Performance Monitoring	August 2010	August 2010
2 nd Quarter Post-Injection Performance Monitoring	November 2010	November 2010
3 rd Quarter Post-Injection Performance Monitoring	February 2011	February 2011
4 th Quarter Post-Injection Performance Monitoring	May 2011	May 2011
SWMU 55		
Phase I Baseline Characterization Event	July 2009	July 2009
Installation of 4 Injection Wells	September 2009	October 2009
Aquifer Slug Testing	October 2009	October 2009
Phase II Baseline Characterization Event	November 2009	November 2009
ISCO Pilot-Scale Test	December 2009	December 2009
Phase III Baseline Characterization Event	February 2010	February 2010
Installation of 4 Monitoring Wells	April 2010	April 2010

SAP Worksheet #16—Project Schedule (continued)

Sampling Event	Start Date	End Date
Phase IV Baseline Characterization Event	April 2010	April 2010
1 st Quarter Post-Injection Performance Monitoring	January 2010	January 2010
2 nd Quarter Post-Injection Performance Monitoring	April 2010	April 2010
3 rd Quarter Post-Injection Performance Monitoring	August 2010	August 2010
Phase V Baseline Characterization Event	October 2010	October 2010

SAP Worksheet #17—Sampling Design and Rationale

SWMU 54

The SWMU 54 TCE pilot test will include installation of monitoring wells, baseline sampling, installation of carbon substrate injection wells, substrate injection, and performance monitoring.

The SWMU 54 benzene pilot-scale test will include installation of monitoring wells, baseline sampling, installation of an AS well, and AS. For the above corrective measure approaches, the rationale for the matrices to be sampled, the number of samples per matrix, the analytical groups, and the concentration levels are discussed in Worksheets #10, 11, 14, and 15. Sample location figures are provided in Appendix A.

SWMU 55

The ISCO pilot test at SWMU 55 will include a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, TOD study, ISCO injections, and quarterly monitoring.

For the above corrective measure approaches, the rationale for the matrices to be sampled, the number of samples per matrix, the analytical groups, and the concentration levels are discussed in Worksheets #10, 11, 14, and 15. Sample location figures are provided in Appendix A.

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SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	No. Samples	Sampling SOP Reference
SWMU 54 Benzene						
54MW01	JM04-54MW01-mmddy	Groundwater	3.9 – 13.9	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW02	JM04-54MW02-mmddy	Groundwater	4.8 – 14.8	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW02	JM04-54MW02-mmddy	Soil	10.5 – 11 and 13.5 - 14	Benzene	2	See Worksheet #14
54MW03	JM04-54MW03-mmddy	Groundwater	4.5-14.5	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW03	JM04-54MW03-mmddy	Soil	10.5 – 11 and 13 – 13.5	Benzene	2	See Worksheet #14

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
54MW04	JM04-54MW04-mmddy	Groundwater	6.3 – 21.3	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW05	JM04-54MW05-mmddy	Groundwater	6.3 – 21.3	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW06	JM04-54MW06-mmddy	Groundwater	5.0 – 15.0	Phase I baseline site characterization sampling event: benzene, dissolved iron, sulfate, sulfide, TOC, nitrate, COD, and alkalinity Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
510DW1	JM04-510DW1-mmddy	Groundwater	19.6 – 24.6	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
510DW2	JM04-510DW2-mmddy	Groundwater	39.2 – 44.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
510MW1	JM04-510MW1-mmddy	Groundwater	3.2 – 13.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
510MW2	JM04-510MW2-mmddy	Groundwater	3.2 – 18.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
510MW3	JM04-510MW3-mmddy	Groundwater	4.9 – 14.9	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
510MW4	JM04-510MW4-mmddy	Groundwater	4.8 – 14.8	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW19	JM04-54MW19-mmddy	Groundwater	15.3 – 25.3	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW20	JM04-54MW20-mmddy	Groundwater	4.9 – 14.9	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW21	JM04-54MW21-mmddy	Groundwater	15.5 – 25.5	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW22	JM04-54MW22-mmddy	Groundwater	5.1 – 15.1	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW23	JM04-54MW23-mmddy	Groundwater	15.2 – 25.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW24	JM04-54MW24-mmddy	Groundwater	5.2 – 15.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW25	JM04-54MW25-mmddy	Groundwater	15.5 – 25.5	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW26	JM04-54MW26-mmddy	Groundwater	4.9 – 14.9	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
54MW27	JM04-54MW27-mmddy	Groundwater	15.2 – 25.2	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW28	JM04-54MW28-mmddy	Groundwater	4.8 – 14.8	Phase II baseline site characterization sampling event: VOCs	1 for each sampling event	See Appendix B
54MW29	JM04-54MW29-mmddy	Groundwater	25.3 – 40.3	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW30	JM04-54MW30-mmddy	Groundwater	14.4 – 24.4	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW31	JM04-54MW31-mmddy	Groundwater	25.1 – 40.1	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW32	JM04-54MW32-mmddy	Groundwater	14.1 – 24.1	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW33	JM04-54MW33-mmddy	Groundwater	25.3 – 40.3	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW34	JM04-54MW34-mmddy	Groundwater	5.1 – 15.1	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW35	JM04-54MW35-mmddy	Groundwater	25.3 – 40.3	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW36	JM04-54MW36-mmddy	Groundwater	15.1 – 25.1	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
54MW37	JM04-54MW37-mmddy	Groundwater	25.2 – 40.2	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW38	JM04-54MW38-mmddy	Groundwater	14.9 – 24.9	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW39	JM04-54MW39-mmddy	Groundwater	14.8 – 24.8	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW40	JM04-54MW40-mmddy	Groundwater	4.8 – 14.8	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW41	JM04-54MW41-mmddy	Groundwater	15.0 – 25.0	Phase III baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW42	JM04-54MW42-mmddy	Groundwater	5.0 – 15.0	Phase IV baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW43	JM04-54MW43-mmddy	Groundwater	5.0 – 15.0	Phase IV baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B
54MW44	JM04-54MW44-mmddy	Groundwater	15.0 – 25.0	Phase IV baseline site characterization sampling event: benzene only	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
SWMU 54 TCE						
54MW07	JM04-54MW07-mmddy	Groundwater	17.7 – 27.7	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, dehalococoides ethenogenes (DHE), and alkalinity	1 for each sampling event	See Appendix B
54MW08	JM04-54MW08-mmddy	Groundwater	16.8 – 26.8	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW09	JM04-54MW09-mmddy	Groundwater	18.4 – 28.4	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW10	JM04-54MW10-mmddy	Groundwater	17.7 – 27.7	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW11	JM04-54MW11-mmddy	Groundwater	17.6 – 27.6	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW12	JM04-54MW12-mmddy	Groundwater	17.8 – 27.8	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
54MW13	JM04-54MW13-mmddy	Groundwater	19.9 – 29.9	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW14	JM04-54MW14-mmddy	Groundwater	17.6 – 27.6	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
510MW5R	JM04-510MW5R-mmddy	Groundwater	17.7 – 27.7	Phase I baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1 for each sampling event	See Appendix B
54MW15	JM04-54MW15-mmddy	Groundwater	17.2 – 27.2	Phase II baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity	1 for each sampling event	See Appendix B
54MW16	JM04-54MW16-mmddy	Groundwater	17.1 – 27.1	Phase II baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity	1 for each sampling event	See Appendix B
54MW17	JM04-54MW17-mmddy	Groundwater	17.4 – 27.4	Phase II baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity	1 for each sampling event	See Appendix B
54MW18	JM04-54MW18-mmddy	Groundwater	17.2 – 27.2	Phase II baseline site characterization and performance monitoring sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, and alkalinity	1 for each sampling event	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
54IW01	JM04-54IW01-mmddy	Groundwater	17.3 – 27.3	Phase II baseline site characterization sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1	See Appendix B
54IW02	JM04-54IW02-mmddy	Groundwater	16.9 – 26.9	Phase II baseline site characterization sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1	See Appendix B
54IW03	JM04-54IW03-mmddy	Groundwater	17.2 - 27.2	Phase II baseline site characterization sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1	See Appendix B
54IW04	JM04-54IW04-mmddy	Groundwater	17.3 – 27.3	Phase II baseline site characterization sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1	See Appendix B
54IW05	JM04-54IW05-mmddy	Groundwater	17.4 – 27.4	Phase II baseline site characterization sampling events: TCE, DCE, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, TOC, MEE, DHE, and alkalinity	1	See Appendix B
SWMU 54 Benzene and TCE Waste Characterization						
NA	JM04-54AQW-mmddy	Aqueous Waste	NA	RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH	1	See Worksheet #14
NA	JM04-54SLW-mmddy	Soil Waste	NA	Toxicity compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH	1	See Worksheet #14

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
SWMU 55						
7MW7	JM04-7MW7-mmddy	Groundwater	10.5 – 25.5	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
7MW10	JM04-7MW10-mmddy	Groundwater	1.8 – 11.8	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
7MW21	JM04-7MW21-mmddy	Groundwater	9.5 – 19.5	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
7MW22	JM04-7MW22-mmddy	Groundwater	11.9 – 21.9	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
7MW23	JM04-7MW23-mmddy	Groundwater	8.5 – 18.5	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
7MW24	JM04-7MW24-mmddy	Groundwater	12.0 – 22.0	Phase I baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW01	JM04-55MW01-mmddy	Groundwater	24.6 – 39.6	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW02	JM04-55MW02-mmddy	Groundwater	9.1 – 24.1	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW03	JM04-55MW03-mmddy	Groundwater	24.2 – 39.2	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW04	JM04-55MW04-mmddy	Groundwater	10.1 – 25.1	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW05	JM04-55MW05-mmddy	Groundwater	25.4 – 40.4	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW06	JM04-55MW06-mmddy	Groundwater	10.0 – 25.0	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW07	JM04-55MW07-mmddy	Groundwater	25.2 – 40.2	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
55MW08	JM04-55MW08-mmddy	Groundwater	10.1 – 25.1	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW09	JM04-55MW09-mmddy	Groundwater	25.1 – 40.1	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW10	JM04-55MW10-mmddy	Groundwater	8.2 – 23.2	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55IW01	JM04-55IW01-mmddy	Groundwater	10.5 – 25.5	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55IW02	JM04-55IW02-mmddy	Groundwater	25.1 – 40.1	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55IW03	JM04-55IW03-mmddy	Groundwater	15.7 – 30.7	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55IW04	JM04-55IW04-mmddy	Groundwater	24.9 – 39.9	Phase II baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55IW01	JM04-55IW01-decomposed-mmddy	Soil	NA	TOD	1	See Worksheet #14
55IW01	JM04-55IW01-lithified-mmddy	Soil	NA	TOD	1	See Worksheet #14
55IW04	JM04-55IW4decomposed-mmddy	Soil	NA	TOD	1	See Worksheet #14
55IW04	JM04-55IW4lithified-mmddy	Soil	NA	TOD	1	See Worksheet #14
55MW11	JM04-55MW11-mmddy	Groundwater	24.3 – 39.3	Phase III baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW12	JM04-55MW12-mmddy	Groundwater	15.1 – 30.1	Phase III baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW13	JM04-55MW13-mmddy	Groundwater	15.4 – 25.4	Phase IV baseline characterization event: TCE	1	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
55MW14	JM04-55MW14-mmddy	Groundwater	25.4 – 40.4	Phase III baseline characterization event and performance monitoring sampling events: TCE	1	See Appendix B
55MW15	JM04-55MW15-mmddy	Groundwater	40.3 – 55.3	Phase III baseline characterization event: TCE	1	See Appendix B
55MW16	JM04-55MW16-mmddy	Groundwater	15.1 – 30.1	Phase III baseline characterization event: TCE	1	See Appendix B
55MW17	JM04-55MW17-mmddy	Groundwater	7.3 – 22.3	Phase III baseline characterization event: TCE	1	See Appendix B
55MW18	JM04-55MW18-mmddy	Groundwater	49.0 – 59.0	Phase III baseline characterization event: TCE	1	See Appendix B
55MW19	JM04-55MW19-mmddy	Groundwater	49.3 – 59.3	Phase III baseline characterization event: TCE	1	See Appendix B
55MW20	JM04-55MW20-mmddy	Groundwater	14.3 – 29.3	Phase III baseline characterization event: TCE	1	See Appendix B
55MW21	JM04-55MW21-mmddy	Groundwater	25.4 – 40.4	Phase IV baseline characterization event: TCE	1	See Appendix B
55MW22	JM04-55MW22-mmddy	Groundwater	52.7 – 67.7	Phase IV baseline characterization event: TCE	1	See Appendix B
55MW23	JM04-55MW23-mmddy	Groundwater	28.7 – 43.7	Phase IV baseline characterization event: TCE	1	See Appendix B
55MW24	JM04-55MW24-mmddy	Groundwater	10.0- 25.0	Phase V baseline characterization event: TCE	1	See Appendix B
55MW25	JM04-55MW25-mmddy	Groundwater	10.0- 25.0	Phase V baseline characterization event: TCE	1	See Appendix B

SAP Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (continued)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	No. Samples	Sampling SOP Reference
SWMU 55 Waste Characterization						
NA	JM04-55AQW-mmddy	Aqueous Waste	NA	RCRA compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH	1	See Worksheet #14
NA	JM04-55SLW-mmddy	Soil Waste	NA	Toxicity compounds (see Worksheet #15 for specific compounds), reactivity (sulfide and cyanide), ignitability, and corrosivity as pH	1	See Worksheet #14

SAP Worksheet #19—Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Container	Sample Volume	Preservation Requirements	Maximum Holding Time
Groundwater	VOCs	SW-846 5030B, 8260B/ GCMSV-003	(3) 40-mL glass	40 mL	pH less than 2 with HCl, Cool to 4°C	14 days
Groundwater	Dissolved Metals	SW-846 3010A, 6010B/ MET-005, MET-010	(1) 250-mL plastic	50 mL	Field filter, Cool to 4°C, pH less than 2 with HNO ₃	180 days
Groundwater	Sulfate, Nitrate	EPA 300.0/WL-042	(1) 250-mL plastic	50 mL	Cool to 4°C	28 days for sulfate; 48 hours for nitrate
Groundwater	Sulfide	SM 4500 S D/WL-033	(1) 1-L plastic	500 mL	Cool to 4°C, pH greater than 9 with NaOH and Zn Acetate	7 days
Groundwater	TOC	SM 5310B/WL-043	(2) 40-mL glass	5 mL	pH less than 2 with HCl, Cool to 4°C	28 days
Groundwater	MEE	RSK-175/GC-024	(2) 40-mL glass	40 mL	Cool to 4°C	14 days
Groundwater	Alkalinity	SM 2320B/WL-063	(1) 250-mL plastic	50 mL	Cool to 4°C	14 days
Groundwater	COD	HACH 8000/WL-021	(1) 50-mL glass	2 mL	pH less than 2 with H ₂ SO ₄ , Cool to 4°C	28 days
Groundwater	Dehalococoides Ethenogenes	q-PCR/MI SOP-q-PCR	Bio-Flo Filter with 1-L volume passed through it or 1-L plastic	1,000 mL	Cool to 4°C	24 hours
Soil	VOCs – Benzene only	SW-846 5035, 8260B/ GCMSV-003	(3) 40-mL glass plus 2-oz jar	5 grams	Cool to 4°C, methanol and DI water	48 hours to preserve/ 14 days analysis
Aqueous	VOCs – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 5030B, 8260B/ GCMSV-003	(3) 40-mL glass	40 mL	pH less than 2 with HCl, Cool to 4°C	14 days
Aqueous	SVOCs – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 3510C, 8270C/ EXT-003, GCMSSV-001	(2) 1-L WMG	1,000 mL	Cool to 4°C	7 days extract/ 40 days analysis

SAP Worksheet #19—Analytical SOP Requirements Table (continued)

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Container	Sample Volume	Preservation Requirements	Maximum Holding Time
Aqueous	Pesticides – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 3510C, 8081B/ EXT-010, GC-013	(2) 1-L WMG	1,000 mL	Cool to 4°C	7 days extract/ 40 days analysis
Aqueous	Herbicides – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 3535A, 8151A/ EXT-017, GC-011	(2) 1-L WMG	1,000 mL	Cool to 4°C	7 days extract/ 40 days analysis
Aqueous	Metals – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 3010A, 6010B, 7470A/MET-005, MET-010, MET-006, MET-008	(1) 250-mL plastic	50 mL	Cool to 4°C, pH less than 2 with HNO ₃	180 days except Hg; 28 days for mercury
Aqueous	PCBs – RCRA compounds (see Worksheet #15 for specific compounds).	SW-846 3510C, 8082A/ EXT-010, GC-023	(2) 1-L WMG	1,000 mL	Cool to 4°C	7 days extract/ 40 days analysis
Aqueous	Ignitability	SW-846 1010A/WL-060	(1) 8-oz glass	50 mL	Cool to 4°C	As soon as possible (ASAP)
Aqueous	Corrosivity	SW-846 9040B/EXT-033	(1) 2-oz glass	50 mL	Cool to 4°C	ASAP
Aqueous	Reactive Cyanide and Sulfide	SW-846 7.3.3.2, 7.3.4.2/ WL-054	(1) 8-oz glass	10 mL	Cool to 4°C	ASAP
Solid/Soil	TCLP – VOC	SW-846 1311, 5030B, 8260B/EXT-026, GCMSV- 003	(1) 4-oz glass	25 grams	Cool 4°C	14 days to TCLP extract/14 days analysis
Solid/Soil	TCLP – Semivolatile Organic Compounds	SW-846 1311, 3510C, 8270C/EXT-026, EXT-003, GCMSSV-001	(1) 8-oz glass	100 grams	Cool 4°C	14 days TCLP extract/7 to prep extract/40 days analysis
Solid/Soil	TCLP – Organochlorine Pesticides	SW-846 1311, 3510C, 8081B/EXT-026, EXT-010, GC-013	(1) 8-oz glass	100 grams	Cool 4°C	14 days TCLP extract /7 to prep extract /40 days analysis
Solid/Soil	TCLP – Herbicides	SW-846 1311, 3535A, 8151A/EXT-026, EXT-017, GC-011	(1) 8-oz glass	100 grams	Cool 4°C	14 days TCLP extract /7 to prep extract /40 days analysis

SAP Worksheet #19—Analytical SOP Requirements Table (continued)

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Container	Sample Volume	Preservation Requirements	Maximum Holding Time
Solid/Soil	TCLP- Metals	SW-846 1311, 3010A, 6010B, 7470A/EXT-026, MET-005, MET-010, MET-006, MET-008	(1) 8-oz glass	100 grams	Cool 4°C	180/180 days ^g ; Hg 28/28 days ^h
Solid/Soil	PCBs	SW-846 3550C, 8082A/EXT-002, GC-023	(1) 8-oz glass	30 grams	Cool 4°C	14 days extract/14 days analysis
Solid/Soil	Corrosivity	SW-846 9045C/EXT-032	(1) 8-oz glass	20 grams	Cool 4°C	Immediate
Solid/Soil	Ignitability	SW-846 1010A/WL-060	(1) 8-oz glass	100 grams	Cool 4°C	Immediate
Solid/Soil	Reactive Cyanide and Sulfide	SW-846 7.3.3.2, 7.3.4.2/WL-054	(1) 8-oz glass	10 grams	Cool 4°C	Immediate

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SAP Worksheet #20—Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
SWMU 55									
Water	VOCs— Trichloroethene only	Phases I through V baseline event = 35 samples	6	5	8 (1 for each day of sample shipment)	4 (1 per day per decontaminated equipment)	8 (1 for each day of sample shipment)	0	66
		Performance Monitoring = 69 (23 samples x 3 events)	9	6	15 (1 for each day of sample shipment)	15 (1 per day per decontaminated equipment)	15 (1 for each day of sample shipment)	0	129
SWMU 54 TCE Plume									
Water	VOCs – Limited List: Trichlorobenzene, cis- 1,2-Dichlorobenzene and Vinyl Chloride	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150
Water	Dissolved Metals-Iron and Manganese only	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Water	Sulfate	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150
Water	Sulfide	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150
Water	TOC	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Water	MEE	Phases I and II baseline event = 18 samples	2	2	2 (1 for each day of sample shipment)	3 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	29
		Performance Monitoring = 78 samples (13 samples x 6 events)	12	6	18 (1 for each day of sample shipment)	18 (1 per day per decontaminated equipment)	18 (1 for each day of sample shipment)	0	150
Water	Alkalinity	Performance Monitoring = 52 samples	8	4	12 (1 for each day of sample shipment)	12 (1 per day per decontaminated equipment)	12 (1 for each day of sample shipment)	0	100
Water	Dehalococcoides ethenogenes	12 (3 samples x 4 events)	0	NA	0	0	NA	0	12
SWMU 54 Benzene Plume									
Water	VOCs – Benzene only	Phases I, III, and IV baseline event = 22 samples	4	3	6 (1 for each day of sample shipment)	6 (1 per day per decontaminated equipment)	6 (1 for each day of sample shipment)	0	47
Water	VOCs	Phase II baseline event = 22 samples	3	2	5 (1 for each day of sample shipment)	5 (1 per day per decontaminated equipment)	5 (1 for each day of sample shipment)	0	42
Water	TCE	Phase III baseline event = 3 samples	1	1	1 (1 for each day of sample shipment)	1 (1 per day per decontaminated equipment)	1 (1 for each day of sample shipment)	0	8

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Water	COD	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	Dissolved Iron	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	Sulfate	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	Sulfide	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	TOC	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	Nitrate	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Water	Alkalinity	Phase I baseline sampling = 6 samples	1	1	2 (1 for each day of sample shipment)	2 (1 per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	14
Soil	VOCs – Benzene only	4 (2 sample near MW02; 2 sample near MW03)	1	1	2 (1 for each day of sample shipment)	2 (1per day per decontaminated equipment)	2 (1 for each day of sample shipment)	0	12

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
SWMUs 54 and 55									
Aqueous	VOCs – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	SVOCs – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Pesticides – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Herbicides – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Metals – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	PCBs – RCRA compounds (see Worksheet #15 for specific compounds).	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Ignitability	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Aqueous	Corrosivity	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Reactive Cyanide	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Aqueous	Reactive Sulfide	7 (3 sample for SWMU 54; 4 sample for SWMU 55)	0	0	0	0	NA	0	7
Solid	TCLP – Volatile Organic Compounds	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	TCLP – Semivolatile Organic Compounds	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	TCLP – Organochlorine Pesticides	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	TCLP – Herbicides	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	TCLP – Metals	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6

SAP Worksheet #20—Field Quality Control Sample Summary Table (continued)

Matrix	Analytical Group	No. of Samples to be Collected	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Blanks	No. of VOC Trip Blanks	No. of Proficiency Testing Samples	Total No. of Samples to Lab
Solid	PCBs	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	Corrosivity	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	Ignitability	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6
Solid	Reactive Cyanide and Sulfide	6 (4 sample for SWMU 54; 2 sample for SWMU 55)	0	0	0	0	NA	0	6

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SAP Worksheet #21—Project Sampling SOP Reference Table

Reference Number	Title, Revision Date, and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
Appendix B for SOP	Monitoring Well Installation	AGVIQ-CH2M HILL	Drill rig	N	Survey of monitoring wells will be conducted as indicated on Worksheet #14.
Appendix B for SOP	Injection Well Installation	AGVIQ-CH2M HILL	Drill rig	N	Survey of injection wells will be conducted as indicated on Worksheet #14.
See Worksheet #18 for groundwater sampling locations and Appendix B for SOPs	Groundwater Sampling	AGVIQ-CH2M HILL	Groundwater sampling pumps and tubing	N	
See Worksheet #18 for soil sampling locations and Appendix B for SOPs	Soil Sampling	AGVIQ-CH2M HILL	Drill rig	N	
See Worksheet #18 for all soil sampling locations and Worksheet #14 for sampling procedures.	Soil Waste Characterization	AGVIQ-CH2M HILL	None	N	
See Worksheet #18 for all soil sampling locations and Worksheet #14 for sampling procedures.	Aqueous Waste Characterization	AGVIQ-CH2M HILL	None	N	
See Appendix B for SOPs	Decontamination of Drilling Rigs and Equipment	AGVIQ-CH2M HILL	Pressure washer	N	
See Appendix B for SOPs	Decontamination of Personnel and Equipment	AGVIQ-CH2M HILL	Decontaminate equipment	N	

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SAP Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Frequency	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
YSI multi-meter	Calibrate probe using multiple Calibration Standard Solutions.	Calibrate mechanical and electronic parts, verify system continuity, check battery, and clean probes. Calibration check.	Visual Inspection	Daily before use, at the end of the day, and when unstable readings occur	Stable readings after 3 minutes pH read 4.0 +/- 3% conductivity reads 4.49 +/- 3%	Clean probe with deionized water and calibrate again. Do not use this instrument if unable to calibrate properly.	FTL
HACH LDO meter	No calibration required.	Recharge battery daily.	Visual Inspection	NA	NA	NA	FTL
PID	Calibrate using ambient air and isobutylene 100-ppm calibration gas.	Recharge battery daily.	Visual Inspection	Daily, before use	Ambient air reads 0.0 ppm +/- 3% Isobutylene gas reads 100 ppm +/- 3%	Follow instructions in manual to clean sensor. Do not use this instrument if unable to calibrate properly.	FTL
FID	Calibrate using ambient air and isobutylene 100-ppm calibration gas.	Recharge battery daily.	Visual Inspection	Daily, before use	Methane gas reads 100 ppm +/- 3%	Follow instructions in manual to clean sensor. Do not use this instrument if unable to calibrate properly.	FTL
Groundwater sampling pumps and tubing	No calibration required. An absorbance testing standard will be purchased from the manufacturer and used daily to ensure instrument is functioning properly.	NA	Inspect pumps, tubing and air/sample line quick-connects	Regularly	Maintained in good working order per manufacturer's recommendations	Replace items.	FTL

SAP Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table (continued)

Field Equipment	Calibration Activity	Maintenance Frequency	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
Colorimeter	No calibration required.	Replace batteries when needed.	Visual Inspection	NA	Potassium permanganate solution must be less than 62.4 mg/L	If solution is greater than 62.4 mg/L, then solution will need to be diluted and reanalyzed.	FTL

SAP Worksheet #23—Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? Y/N)
GCMSV-003	SOPs for the Analysis of Volatile Mass Spec Samples Method 8260B, Revision 16	Definitive	Groundwater, Soil, Soil TCLP Extract/ Liquid Waste Characterization	Agilent 6890 or 7890 GC with a 5973 or 5975 Mass Spectrometer	GCAL	N
MET-010	SOP for Analysis of Samples by ICP, Revision 16	Definitive	Groundwater	Perkin Elmer 5300DV or 4300DV ICP	GCAL	N
WL-042	SOP for IC, Revision 12	Definitive	Groundwater	Dionex Series 500i Ion Chromatograph	GCAL	N
WL-033	SOP for Sulfide, Revision 5	Definitive	Groundwater	HACH 2800 Spectrophotometer	GCAL	N
WL-043	SOP for TOC, Revision 7	Definitive	Groundwater	Shimadzu TOC-5050	GCAL	N
GC-024	SOP for the Analysis of Dissolved Gas in Groundwater, Revision 5	Definitive	Groundwater	HP 5890 Series II GC	GCAL	N
WL-063	SOP for Automated Analysis of Alkalinity, Revision 5	Definitive	Groundwater	Mettler Toledo DL53 Autotitrator	GCAL	N
WL-021	SOP for COD, Revision 8	Definitive	Groundwater	HACH 2800 Spectrophotometer	GCAL	N
MI SOP-qPCR	Quantitative Polymerase Chain Reaction (qPCR) revision date 5/22/09	Definitive	Groundwater	ABI 7300	Microbial Insights, Inc.	N

SAP Worksheet #23—Analytical SOP References Table (continued)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? Y/N)
EXT-026	SOP for Toxicity Characteristic Leaching Procedure – Method 1311, Revision 7	Definitive	Soil TCLP Extract	NA	GCAL	N
EXT-003	SOP for Base/Neutral/Acid Sample Extraction Using Separatory Funnel, Revision 15	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	NA	GCAL	N
GCMSV-001	SOP for the Analysis of Semi-volatile Mass Spec Samples for 8270C, Revision 14	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	Agilent/5973-6890N or 5975-6890N GC/MS	GCAL	N
EXT-010	SOP for Preparation of Pesticide/PCB Sample Extraction Using Separatory Funnel, Revision 12	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	NA	GCAL	N
GC-013	SOP for Pesticides – Method 8081B, Revision 12	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	Agilent 6890N GC/ECD	GCAL	N
EXT-017	SOP for Preparation of Aqueous Samples for Herbicides, Revision 19	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	NA	GCAL	N

SAP Worksheet #23—Analytical SOP References Table (continued)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? Y/N
GC-011	SOP for Chlorinated Herbicides 8151A, Revision 7	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	Agilent 6890N GC/ECD	GCAL	N
MET-005	SOP for ICP Water Preparation, Revision 12	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	NA	GCAL	N
MET-010	SOP for Analysis of Samples by ICP, Revision 16	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	Perkin Elmer 5300DV or 4300DV ICP	GCAL	N
MET-006	SOP for Sample Preparation – Mercury, Revision 17	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	NA	GCAL	N
MET-008	SOP for Mercury Analysis, Revision 14	Definitive	Soil TCLP Extract/ Liquid Waste Characterization	Perkin Elmer FIMS 400 Mercury Analyzer	GCAL	N
EXT-002	SOP for Preparation of Pesticide/PCB Low Level Soil/Sediment Samples, Revision 13	Definitive	Soil Waste Characterization	NA	GCAL	N
GC-023	SOP for PCB – Method 8082A, Revision 8	Definitive	Soil Waste Characterization/ Liquid Waste Characterization	Agilent 6890N GC/ECD	GCAL	N
EXT-032	SOP for Determining pH in Solid or Waste Samples, Revision 8	Definitive	Soil Waste Characterization	Orion SA720 pH Meter	GCAL	N

SAP Worksheet #23—Analytical SOP References Table (continued)

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? Y/N)
EXT-033	SOP for Determining pH in Water Samples, Revision 7	Definitive	Liquid Waste Characterization	Orion SA720 pH Meter	GCAL	N
WL-060	SOP for Flashpoint – Automated, Revision 3	Definitive	Soil Waste Characterization/ Liquid Waste Characterization	Herzog MP-330- Automated Pensky Marten Closed Cup Flashpoint Tester	GCAL	N
WL-054	SOP for Reactive Cyanide and Reactive Sulfide, Revision 7	Definitive	Soil Waste Characterization/ Liquid Waste Characterization	NA	GCAL	N
SAD-001	SOP for Log-In, Revision 13	NA	Groundwater/Soil/ Soil Waste Characterization/ Liquid Waste Characterization	NA	GCAL	N
GEN-009	SOP for Waste Collection, Storage, and Disposal, Revision 5	NA	Groundwater/Soil/ Soil Waste Characterization/ Liquid Waste Characterization	NA	GCAL	N

SAP Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
GCMS – VOCs	Initial Calibration (ICAL) – Minimum five-point calibration for all analytes	Initial calibration prior to sample analysis	Relative standard deviation (RSD) less than or equal to 30 for RFs of the CCCs; Average %RSD less than or equal to 15% for all compounds	Repeat calibration if criterion is not met.	Analyst, Supervisor	GCAL SOP GCMSV-003
	Second source calibration verification	Once after each initial calibration	All analytes within $\pm 25\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
	Calibration verification (CV)	CV daily, before sample analysis, and every 12 hours of analysis time	CCCs less than or equal to 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
GCMS Semivolatiles	ICAL – Minimum five-point calibration for all analytes	Initial calibration prior to sample analysis	RSD less than or equal to 30 for RFs of the CCCs; Average %RSD less than or equal to 15% for all compounds	Repeat calibration if criterion is not met.	Analyst, Supervisor	GCAL SOP GCMSV-001
	Second source calibration verification	Once after each initial calibration	All analytes within $\pm 25\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
	CV	CV daily, before sample analysis, and every 12 hours of analysis time	CCCs less than or equal to 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	

SAP Worksheet #24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
Pesticides/PCBs Herbicides GC/ECD	ICAL – Minimum five-point calibration for all analytes	Initial calibration prior to sample analysis	RSD less than or equal to 20% for all compounds or linear or quadratic calibration correlation coefficient greater than 0.990	Repeat calibration if criterion is not met.	Analyst, Supervisor	GCAL SOP GC-013 GC-023 GC-011
	Second source calibration verification	Once after each initial calibration	All analytes within $\pm 15\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
	CCV	CCV after every 10 samples and at the end of the analytical sequence	%D or Drift less than 20% for all analytes	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
RSK-175 GC FID	ICAL – Minimum five-point calibration for all analytes	Initial calibration prior to sample analysis	RSD less than or equal to 25% for all compounds or linear or quadratic calibration correlation coefficient greater than 0.990	Repeat calibration if criterion is not met.	Analyst, Supervisor	GCAL SOP GC-024
	Second source calibration verification	Once after each initial calibration	All analytes within $\pm 25\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
	CV	CV daily, before sample analysis, and every 12 hours of analysis time	%D or Drift less than 20% for all analytes	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
ICP – Metals	ICAL – Minimum one standard and a calibration blank	Daily initial calibration prior to sample analysis	No acceptance criteria for blank and one standard	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP MET-010

SAP Worksheet #24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
ICP – Metals	Initial calibration verification (ICV)	Once after each initial calibration	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	GCAL SOP MET-010
	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
IC – Anions	ICAL – Minimum three standards and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient less than or equal to 0.995 for linear regression	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP WL-042
	ICV	Once after each initial calibration	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
	CCV	CCV after every 10 samples and at the end of the analytical sequence	All analytes within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	

SAP Worksheet #24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
COD, Sulfide – HACH 2800 Spectrophotometer	ICAL – Minimum three standards and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient greater than or equal to 0.995 for linear regression	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP WL-033
	ICV	Once after each initial calibration	Analyte within $\pm 10\%$ of expected value	Repeat initial calibration.	Analyst, Supervisor	
	CCV	CCV after every 10 samples and at the end of the analytical sequence	Analyte within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
TOC – Shimadzu TOC 5050 Analyzer	ICAL – Minimum three standards and a calibration blank for TC and IC	Initial calibration prior to sample analysis	Correlation coefficient greater than or equal to 0.995 for linear regression for TC and IC	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP WL-043
	ICV for TC and IC	Once after each initial calibration	Analyte within $\pm 10\%$ of expected value for TC and IC	Repeat initial calibration.	Analyst, Supervisor	
	CCV	CCV after every 10 samples and at the end of the analytical sequence	Analyte within $\pm 10\%$ of expected value for TC and IC	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
Alkalinity – Mettler Toledo DL53 Autotitrator	Calibration of pH buffers 4, 7, and 10	Daily before use	Slope is -52 to-65	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP WL-063

SAP Worksheet #24—Analytical Instrument Calibration Table (continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
Corrosivity (pH) Orion SA720 pH Meter	Calibration of pH buffers 4, 7, and 10	Daily before use	QC check of mid-range buffer must be within 0.05 pH units of the true value.	Recalibrate and/or perform necessary equipment maintenance.	Analyst, Supervisor	GCAL SOP EXT-032 EXT-033
Ignitibility – Closed Cup Flashpoint Tester	NA	NA	NA	NA	NA	GCAL SOP WL-066
Reactivity Cyanide – Lachat	ICAL – Minimum three standards and a calibration blank	Initial calibration prior to sample analysis	Correlation coefficient greater than or equal to 0.995 for linear regression	Repeat initial calibration.	Analyst, Supervisor	GCAL SOP WL-015
	ICV	Once after each initial calibration	Analyte within $\pm 10\%$ of expected value	Repeat initial calibration.	Analyst, Supervisor	
	CCV	CCV after every 10 samples and at the end of the analytical sequence	Analyte within $\pm 10\%$ of expected value	Repeat initial calibration and reanalyze all samples analyzed since the last successful CV.	Analyst, Supervisor	
Reactivity Sulfide-Titration (manual)	NA	NA	NA	NA	NA	GCAL SOP WL-051
Dehalococcoides Ethenogenes- ABI 7300	CCV	Primary – annual	Standard curve correlation coefficient greater than 0.95	Rerun assay/check reagents.	Anita Biernacki/ Microbial Insights	MI SOP q-PCR
	CV	Secondary – every plate (assay)	CT value within 2 units of same point on standard curve	Reprocess the sample until it meets criteria.	Anita Biernacki/ Microbial Insights	

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SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person(s)	SOP Reference
Gas Chromatograph / Mass Spectrometer (GC/MS)	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner	Volatiles	Monitor instrument performance via CCV	As needed	No maintenance is required as long as instrument QC meets DOD 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
GCMS -	Clean Injection port and replace liner; clip column; leak check; maintain pumps by checking replacing pump oil	Semi-volatiles	Monitor instrument performance via CCV DFTPP tune, breakdown and tailing	Daily	No maintenance is required as long as instrument QC meets DOD criteria	Change column; clean source	Analyst, Supervisor	GCAL SOP GCMSSV-001
GC ECD	Clean injection port and replace liner; clip column; maintain pumps; ECD wipe test	Pest/PCBs/Herb	Monitor instrument performance via CCV Monitor DDT and Endrin breakdown for 8081/8082	Daily; wipe test annually	Breakdown less than 15%, calibration and QC criteria met	Change column; instrument maintenance	Analyst, Supervisor	GCAL SOP GC-013, GC-023, GC-011
GC/FID	Change septa and liner	Dissolved MEE	Monitor instrument performance via CCV	Check septa at least daily; change liner every three batches	Calibration and QC criteria met	Change column; instrument maintenance	Analyst, Supervisor	GCAL SOP GC-024
ICP - Metals	Perform leak test, change pump tubing, change torch and window, clean filters	TAL Metals	Monitor instrument performance via CCV and CCBlank	As needed	No maintenance is required as long as instrument QC meets DOD criteria	Change pump tubing, change torch and window, clean filters; recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP MET-010
FIMS 400 Mercury Analyzer	Change pump tubing, clean optical cell and lenses, replace mercury lamp	Mercury	Check pump tubing, monitor absorbance of standards	As needed	QC meets DOD acceptance criteria	Clean and replace parts as needed; recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP MET-008
IC - Anions	Prime pump, change column	Sulfate and Nitrate	Monitor instrument performance via CCV and CCBlank	As needed	No maintenance is required as long as instrument QC meets DOD criteria	Change column; recalibrate and reanalyze affected data	Analyst, Supervisor	GCAL SOP WL-042
HACH 2800 Spectrophotometer	Inspect cell; lamp maintenance	Sulfide	Monitor instrument performance via CCV and CCBlank	As needed	No instrument error message	Clean or replace as necessary	Analyst, Supervisor	GCAL SOP WL-033
Shimadzu TOC 5050 Analyzer	Injection port; injection needle; catalyst	TOC	Monitor instrument performance via CCV and CCBlank	Daily with loss of sensitivity or lack of response	QC meets DOD acceptance criteria	Replace or clean as needed	Analyst, Supervisor	GCAL SOP WL043
Mettler Toledo DL53 Autotitrator	Burette drive	Alkalinity	Calibration check performed by contractor	Annual	5,000 to 1 accuracy to volume ratio	Instrument maintenance	Performed by contractor	GCAL SOP WL-063

SAP Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (continued)

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person(s)	SOP Reference
HACH 2800 Spectrophotometer	Inspect cell; lamp maintenance	COD	Check thermometer against NIST thermometer	Quarterly	No more than 2°C correction	Place correction factor on digestion block or replace	QA	GCAL SOP WL-021
Orion SA720 pH Meter	Check electrode	Corrosivity (pH); calibration check	Flush and refill electrode; clean electrode with methanol	As needed	No instrument error message	Clean or replace as necessary	Analyst, Supervisor	GCAL SOP EXT-032, EXT-033
Closed Cup Flashpoint Tester – Ignitability	Hood flow; torch temperature	Ignitability	Check hood flow and temp	Each day of use	Air flow 140 feet/minute Temperature greater than 1,000°C	Adjust sash for air flow; adjust or replace torch	Analyst, Supervisor	GCAL SOP WL-066
LACHAT	Clean probe and colorimeter filters	Reactivity Cyanide	Leak check; tubing	With loss of sensitivity or erratic response; inspect tubing and filters daily	QC meets DOD acceptance criteria	Clean and replace parts as needed	Analyst, Supervisor	GCAL SOP WL-015
Titration	NA	Reactivity Sulfide	NA	NA	NA	NA	NA	NA
ABI 7300	Manufacturer Maintenance Service Contract	Dehalococoides ethenogens	Pure spectra calibration; region of interest calibration; SDS software update; inspection and thorough cleaning of instrument	Twice a year	As defined by ABI 7300 Sequence Detection System and ROI calibration	Rerun calibration until it fits into specifications.	Manufacturer	MI SOP-qPCR

SAP Worksheet #26—Sample Handling System

Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): Andrew O'Connor/AGVIQ-CH2M HILL
Sample Packaging (Personnel/Organization): Andrew O'Connor/AGVIQ-CH2M HILL
Coordination of Shipment (Personnel/Organization): Andrew O'Connor/AGVIQ-CH2M HILL
Type of Shipment/Carrier: Overnight/FedEx
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Michelle Raborn/GCAL
Sample Custody and Storage (Personnel/Organization): Michelle Raborn/GCAL
Sample Preparation (Personnel/Organization): John Bailey/GCAL
Sample Determinative Analysis (Personnel/Organization): Mark Peterman/GCAL
Sample Archiving
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days from receipt
Biological Sample Storage (No. of days from sample collection): NA
Sample Disposal
Personnel/Organization: Obbie Tillotson/GCAL
Number of Days from Analysis: 60 days from receipt

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SAP Worksheet #27—Sample Custody Requirements Table

Sample Labeling

Sample labels will include, at a minimum, client name, site, sample ID, date/time collected, analysis group or method, preservative, and sampler's initials. Labels will be taped to the jar to ensure they do not separate.

Field Sample Custody Procedures (Sample Collection, Packaging, Shipment, and Delivery to Laboratory)

Samples will be collected by field team members under the supervision of the field team leader. As samples are collected, they will be placed into containers and labeled, as outlined above. Samples will be cushioned with packaging material and placed into coolers containing enough ice to keep the samples below 4°C until they are received by the laboratory. The chain-of-custody will also be placed into the cooler. Coolers will be shipped to the laboratory via FedEx, with the air bill number indicated on the chain-of-custody (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples.

Laboratory Sample Custody Procedures (Receipt of Samples, Archiving, Disposal)

See the laboratory sample handling and disposal SOPs: GCAL SOP SAD-001 and GCAL SOP GEN 009.

Sample Identification Procedures

Upon opening the cooler, the receiving clerk signs the chain-of-custody and then takes the temperature using the temperature blank (if absent, then a sample container or infrared thermometer is used). The sample containers in the cooler are unpacked and checked against the client's chain-of-custody and any discrepancies or breakage is noted on the chain-of-custody. Next, if any water samples require preservative, the clerk will check the pH values to see if they are in the acceptable pH range. The clerk will deliver the chain-of-custody (and any other paperwork; e.g. temperature or pH QA notice) to the project manager for Laboratory Information Management System (LIMS) entry and client contact (if needed).

The field logbook will identify the sample ID with the location, depth, date/time collected, and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the chain-of-custody. The laboratory will send sample log-in forms to the EIS to check sample IDs and parameters are correct.

Chain-of-Custody Procedures

Chains-of-custody will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID, date/time collected, number and type of containers, preservative information, analysis method, and comments. The chain-of-custody will also have the sampler's name and signature. The chain-of-custody will link location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the LIMS database for each sample.

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SAP Worksheet #28—Laboratory QC Samples Table

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix: Groundwater, Soil Analytical Group: VOCs Analytical Method/SOP Reference: SW-846 8260B/GCAL SOP GCMSV-003						
Internal standards verification	In all field samples and standards; four internal standards per sample.	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL. EICP area within -50% to $+100\%$ of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalyze samples with IS failures.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL. EICP area within -50% to $+100\%$ of ICAL midpoint standard.
Method blank	One per batch	No analytes detected greater than $\frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected greater than RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than $\frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
Laboratory Control Sample (LCS)	One per batch	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3.0	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3
MS	One per batch per matrix	Same criteria as LCS	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Same criteria as LCS
MSD	One per batch per matrix	RPD less than or equal to 30%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	RPD less than or equal to 30%
Surrogate spike	In all field and QC samples; four surrogates per sample for waters, two surrogates per sample for soils.	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3.0	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3
Matrix: Groundwater Analytical Group: Metals Analytical Method/SOP Reference: SW-846 6010 B/GCAL SOP MET-010						
Method blank	One per preparatory batch	No analytes detected greater than $\frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected greater than RL.	Correct problem; reprep and reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than $\frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per preparatory batch	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3.0	Correct problem; reprep and reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3
Dilution test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within $\pm 10\%$ of the original determination.	Perform post-digestion spike (PDS) addition	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Five-fold dilution must agree within $\pm 10\%$ of the original determination.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples is less than 50 times the MDL	Recovery within 75-125% of expected result.	Flag data.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery within 75-125% of expected result.

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
MS	One per preparatory batch per matrix	Same criteria as LCS	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Same criteria as LCS
MSD or sample duplicate	One per preparatory batch per matrix	RPD less than or equal to 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	RPD less than 20%
Matrix: Groundwater Analytical Group: Anions Analytical Method/SOP Reference: EPA 300.0/GCAL SOP WL-042						
Method blank	One per batch of 20 or fewer samples	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than RL.	Correct problem; reprep and reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch of 20 or fewer samples	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3.0	Correct problem; reprep and reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	QC acceptance criteria as specified Appendix DOD-D of the DOD QSM Version 3
MS	One per batch of 20 or fewer samples	Same criteria as LCS	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Same criteria as LCS
MSD	One per batch of 20 or fewer samples	RPD less than or equal to 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	RPD less than or equal to 20%
Sample duplicate	One per every 10 samples	%D less than or equal to 10%	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	%D less than or equal to 10%
Matrix: Groundwater Analytical Group: Sulfide Analytical Method/SOP Reference: SM 4500 S D/GCAL SOP WL-033						
Method blank	One per batch of 20 or fewer samples	No analytes detected greater than ½ the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL.
LCS	One per batch of 20 or fewer samples	Recovery 80-120%	Correct problem; reanalyze any sample associated with an LCS that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 80-120%
MS	One per batch of 20 or fewer samples	Recovery 75-125%	Report data with a narrative stating the sample is affected by a matrix interference.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 75-125%
Sample duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 25% for concentrations greater than 5 times the RL	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 25% for concentrations greater than 5 times the RL
Matrix: Groundwater Analytical Group: TOC Analytical Method/SOP Reference: SM 5310B/GCAL SOP WL-043						

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method blank	One per batch of 20 or fewer samples	No analytes detected greater than ½ the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL.
LCS	One per batch of 20 or fewer samples	Recovery 80-120%	Correct problem; reanalyze any sample associated with an LCS that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 80-120%
MS	One per batch of 10 or fewer samples	Recovery 75-125%	Report data with a narrative stating the sample is affected by a matrix interference.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 75-125%
Sample duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 25% for concentrations greater than 5 times the RL	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 25% for concentrations greater than 5 times the RL
Matrix: Groundwater Analytical Group: COD Analytical Method/SOP Reference: HACH 8000/GCAL SOP WL-021						
Method blank	One per batch of 20 or fewer samples	No analytes detected greater than ½ the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL.
LCS	One per batch of 20 or fewer samples	Recovery 80-120%	Correct problem; reanalyze any sample associated with an LCS that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 80-120%
MS	One per batch of 20 or fewer samples	Recovery 75-125%	Report data with a narrative stating the sample is affected by a matrix interference.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 75-125%
Sample duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 25% for concentrations greater than 5 times the RL	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 25% for concentrations greater than 5 times the RL
Matrix: Groundwater Analytical Group: Alkalinity Analytical Method/SOP Reference: SM 2320B/GCAL SOP WL-063						
LCS	One per batch of 20 or fewer samples	Recovery 90-110%	Correct problem; reanalyze any sample associated with an LCS that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 90-110%
Sample duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 10% for concentrations greater than 5 times the RL	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 10% for concentrations greater than 5 times the RL
Matrix: Groundwater Analytical Group: MEE Analytical Method/SOP Reference: RSK-175/GCAL SOP GC-024						
Method blank	One per batch	No analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than the RL.

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch	Recovery 30-170%	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery 30-170%
MS	One per batch per matrix	Same criteria as LCS	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Same criteria as LCS.
MSD	One per batch per matrix	RPD less than or equal to 20%	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	RPD less than or equal to 20%
Surrogate spike	In all field and QC samples	Surrogate Recovery 40-160%	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	Surrogate Recovery 40-160%
Matrix: Groundwater Analytical Group: Microbiological Analytical Method/SOP Reference: qPCR/MI SOP qPCR						
Positive control	1 per analytical assay plate	CT value within 2 units of same point on standard curve	Rerun assay/check reagents.	Anita Biernacki	Bias Contamination	CT value within 2 units of same point on standard curve.
Negative control	1 per analytical assay plate	values for positive samples are set above any fluorescence for the negative control	Rerun assay; may have to reoptimize assay.	Anita Biernacki	Bias Contamination	Values for positive samples are set above any fluorescence for the negative control.
Matrix: Aqueous Waste/Solid Waste Analytical Group: Total (Aq.)/TCLP (SW) VOCs – RCRA Compounds Only Analytical Method/SOP Reference: SW-846 8260B/GCAL SOP GCMSV-003						
Internal standards verification	In all field samples and standards; four internal standards per sample.	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalyze samples with IS failures.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time \pm 30 seconds from retention time of the midpoint standard in the ICAL. EICP area within -50% to +100% of ICAL midpoint standard.
Method blank	One per batch	No analytes detected greater than 1/2 the RL. For common laboratory contaminants, no analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than 1/2 the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch	Laboratory limits.	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MS	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MSD	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	Laboratory limits

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogate spike	In all field and QC samples; four surrogates per sample.	Laboratory limits.	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
Matrix: Aqueous Waste/Solid Waste Analytical Group: Total (Aq.)/TCLP (SW) SVOCs – RCRA Compounds Only Analytical Method/SOP Reference: SW-846 8270C/GCAL SOP GCMSSV-001						
Internal standards verification	In all field samples and standards; six internal standards per sample.	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 mass spectrometer and GC for malfunctions. Reanalyze samples with IS failures.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within –50% to +100% of ICAL midpoint standard.
Method blank	One per batch	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch	Laboratory limits.	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MS	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MSD	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	Laboratory limits
Surrogate spike	In all field and QC samples; six surrogates per sample.	Laboratory limits.	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
Matrix: Aqueous Waste/Solid Waste Analytical Group: Total (Aq.)/TCLP (SW) Pesticides/Herbicides – RCRA Compounds Only Analytical Method/SOP Reference: SW-846 8081B/8151A/GCAL SOP GC-013/GC-011						
Method blank	One per batch	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch	Laboratory limits.	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MS	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
MSD	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	Laboratory limits

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Surrogate spike	In all field and QC samples; two surrogates per sample for pesticides and one surrogate per sample for herbicides.	Laboratory limits.	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits
Matrix: Aqueous Waste/Solid Waste Analytical Group: PCBs Analytical Method/SOP Reference: SW-846 8082A/GCAL SOP GC-023						
Method blank	One per batch	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch	Laboratory limits.	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
MS	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
MSD	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	Laboratory limits.
Surrogate spike	In all field and QC samples; one surrogate per sample.	Laboratory limits.	Correct problem; reanalyze all failed samples for failed surrogates if sufficient sample is available.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
Matrix: Aqueous Waste/Solid Waste Analytical Group: Total (Aq.)/TCLP (SW) Metals – RCRA Compounds Only Analytical Method/SOP Reference: SW-846 6010B/7470A/GCAL SOP MET-010/MET-008						
Method blank	One per batch	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.	Correct problem; reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than ½ the RL. For common laboratory contaminants, no analytes detected greater than the RL.
LCS	One per batch	Laboratory limits.	Correct problem; reanalyze all samples in the associated batch for failed analytes.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
MS	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
MSD	One per batch per matrix	Laboratory limits.	Contact the client to determine if additional measures are required.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	Laboratory limits.
Dilution test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	Perform PDS addition	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Five-fold dilution must agree within ± 10% of the original determination

SAP Worksheet #28—Laboratory QC Samples Table (continued)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
PDS addition	When dilution test fails or analyte concentration in all samples is less than 50 times the MDL	Recovery within 75-125% of expected result.	Flag data.	Analyst, Supervisor, QA Manager	Accuracy Bias	Recovery within 75-125% of expected result.
Matrix: Aqueous Waste/Solid Waste Analytical Group: Reactivity Cyanide, Sulfide Analytical Method/SOP Reference: SW-846 Ch.7/GCAL SOP WL-054						
Method blank	One per batch of 20 or fewer samples	No analytes detected greater than the RL.	Correct problem; reprep and reanalyze any sample associated with a blank that fails criteria, except when the sample analysis resulted in a non-detect.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected greater than the RL.
LCS	One per batch of 20 or fewer samples	Laboratory limits.	Correct problem; reanalyze any sample associated with an LCS that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	Laboratory limits.
Sample duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 25% for concentrations greater than 5 times the RL	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 25% for concentrations greater than 5 times the RL
Matrix: Aqueous Waste/Solid Waste Analytical Group: Ignitability Analytical Method/SOP Reference: SW-846 1010A/GCAL SOP WL-060						
LCS (o-Xylene)	One per batch of 20 or fewer samples	Flashpoint 90 ± 2°F	Do not analyze samples without a daily LCS which meets criteria.	Analyst, Supervisor, QA Manager	Accuracy	Flashpoint 90 ± 2°F
Matrix: Aqueous Waste/Solid Waste Analytical Group: Corrosivity (pH) Analytical Method/SOP Reference: SW-846 9040B/9045C/GCAL SOP EXT-033/EXT-032						
Sample Duplicate	One per batch of 20 or fewer samples	RPD less than or equal to 5%	Correct problem and reanalyze sample and duplicate.	Analyst, Supervisor, QA Manager	Precision	RPD less than or equal to 5%
QC check	Analyze a mid-range buffer as a QC check after calibration	The determined pH must be within +/- 0.05 pH units	Correct problem; recalibrate and reanalyze any sample associated with QC that fails criteria.	Analyst, Supervisor, QA Manager	Accuracy Bias	The determined pH must be within +/- 0.05 pH units

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SAP Worksheet #29—Project Documents and Records Table

Document	Where Maintained
Field Notebooks	Electronic .pdf copies in the project file. Hardcopy (bound notebook) in the project file. Archived at project closeout ^a .
Chain-of-Custody Records	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Air Bills	Hardcopy in the project file. Archived at project closeout.
Telephone Logs	Hardcopy in the project file. Archived at project closeout.
Corrective Action Forms	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Electronic Field Data Deliverables	Loaded in the Field Database then transferred to the SQL Data Warehouse as the final repository.
Various field measurements	Recorded in Field Notebook and stored in SQL Data Warehouse.
All field equipment calibration information	Recorded in Field Notebook.
Pertinent telephone conversations	Recorded in Field Notebook.
Field equipment maintenance records	Inspected by Field Team Leader. Not maintained.
Sample Receipt, Custody, and Tracking Records	Electronic .pdf copies in the project file. Hardcopy in the full data package and stored in SQL sample tracking database.
Standard Traceability Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Calibration Logs	Hardcopy in the full data package. Archived at project closeout.
Sample Prep Logs	Hardcopy in the full data package. Archived at project closeout.
Run Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Maintenance, Testing, and Inspection Logs	Hardcopy in the full data package. Archived at project closeout.
Reported Field Sample Results	Electronic .pdf copies in the project file. Hardcopy in the data package. Archived at project closeout.
Reported Results for Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.

SAP Worksheet #29—Project Documents and Records Table (continued)

Document	Where Maintained
Instrument Printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Data Package Completeness Checklists	Hardcopy in the data validation report. Archived at project closeout.
Sample Disposal Records	Maintained by the laboratory.
Extraction/Cleanup Records	Maintained by the laboratory.
Raw Data	Hardcopy in the full data package. Archived at project closeout. Hard copies at Iron Mountain and DVD/CD backups onsite at AGVIQ-CH2M HILL.
Field Sampling Audit Checklists	Hardcopy in the project file. Archived at project closeout.
Fixed Laboratory Audit Checklists	If completed, hardcopy in the project file. Archived at project closeout.
Data Validation Reports	Electronic .pdf copies in the project file. Hardcopy stored with the data package. Archived at project closeout. Hard copies at Iron Mountain and DVD/CD backups onsite at AGVIQ-CH2M HILL.
Electronic Data Deliverables and Electronic Login Deliverables	EDDs are loaded into the SQL Data Warehouse as the final repository. The ELDs are loaded into the SQL Sample Tracking DB as the final repository.
<p>Notes:</p> <p>^a Data archiving will be done in accordance with Navy requirements. AGVIQ-CH2M HILL will provide the Navy (currently Bonnie Capito) all data and reports for archiving.</p>	

SAP Worksheet #30—Analytical Services Table

All samples will be shipped to GCAL, in Baton Rouge, Louisiana for analysis. The data package will include hardcopy data (including raw data), a CD-ROM containing portable document format (PDF) versions of the data package, and an electronic data deliverable. The data package will be due within 14 calendar days of sample receipt.

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Groundwater	VOCs	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	SW-846 5030B, 8260B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	Dissolved Metals	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	SW-846 3010A, 6010B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	Sulfate, Nitrate	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	EPA 300.0	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	Sulfide	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	SM 4500 S D	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	TOC	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	SM 5310B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD

SAP Worksheet #30—Analytical Services Table (continued)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Groundwater	MEE	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	RSK-175	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	Alkalinity	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	SM 2320B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	COD	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	HACH 8000	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Groundwater	Dehalococcoides Ethenogenes	See Worksheet #18 and Figures 4, 5, and 6 for monitoring or injection well locations.	q-PCR	14 days	Microbial Insights, Inc. 2340 Stock Creek Blvd. Rockford, TN 37853-3044 Phone: (865) 573-8188	TBD
Soil	VOCs-Benzene only	See Worksheet #18.	SW-846 5035, 8260B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD

SAP Worksheet #30—Analytical Services Table (continued)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Aqueous Waste	VOCs – RCRA compounds (see Worksheet #15 for specific compounds).	See Worksheet #18.	SW-846 5030B, 8260B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	SVOCs – RCRA compounds (see Worksheet #15 for specific compounds).	See Worksheet #18.	SW-846 3510C, 8270 C	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	Pesticides/PCBs – RCRA compounds (see Worksheet #15 for specific compounds).	See Worksheet #18.	SW-846 3510C, 8081B/8082 A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	Herbicides – RCRA compounds (see Worksheet #15 for specific compounds).	See Worksheet #18.	SW-846 3535A, 8151A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	Metals – RCRA compounds (see Worksheet #15 for specific compounds).	See Worksheet #18.	SW-846 3010A, 6010B, 7470A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	Corrosivity	See Worksheet #18.	SW-846 9040B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD

SAP Worksheet #30—Analytical Services Table (continued)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Aqueous Waste	Ignitability	See Worksheet #18.	SW-846 1010A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Aqueous Waste	Reactive Cyanide/Sulfide	See Worksheet #18.	SW-846 7.3.3.2, 7.3.4.2	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	TCLP VOCs	See Worksheet #18.	SW-846 1311, 5030B, 8260B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	TCLP SVOCs	See Worksheet #18.	SW-846 1311, 3510C, 8270C	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	TCLP Pesticides	See Worksheet #18.	SW-846 1311, 3510C, 8081B	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	TCLP Herbicides	See Worksheet #18.	SW-846 1311, 3535A, 8151A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD

SAP Worksheet #30—Analytical Services Table (continued)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Solid Waste	TCLP Metals	See Worksheet #18.	SW-846 1311, 3010A, 6010B, 7470A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	Total PCBs	See Worksheet #18.	SW-846 3550C, 8082A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	Corrosivity	See Worksheet #18.	SW-846 9045C	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	Ignitability	See Worksheet #18.	SW-846 1010A	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD
Solid Waste	Reactive Cyanide/Sulfide	See Worksheet #18.	SW-846 7.3.3.2, 7.3.4.2	14 days	GCAL 7979 GSRI Ave. Baton Rouge, LA 70820 Contact: Shelley Bourgeois Phone: 225-769-4900	TBD

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SAP Worksheet #31—Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing CA	Person(s) Responsible for Monitoring Effectiveness of CA
Field Performance Audit	One during sampling activities	Internal	AGVIQ-CH2M HILL	Alicia Nobles, QC Manager AGVIQ-CH2M HILL	Project Field Team AGVIQ-CH2M HILL	Tom Beisel, PM AGVIQ-CH2M HILL	Theresa Rojas, Program QA/QC Manager AGVIQ-CH2M HILL
Safe Work Observation	One per week during field activities	Internal	AGVIQ-CH2M HILL	Andrew O'Connor, SSC AGVIQ-CH2M HILL	Project Field Team AGVIQ-CH2M HILL	Mike Goldman, H&S Officer AGVIQ-CH2M HILL	Andrew O'Connor, SSC AGVIQ-CH2M HILL
Offsite Laboratory Technical Systems Audit	Laboratory must have current Naval Facilities Engineering Service Center (NFESC) evaluation letter which will identify the period of performance. The laboratory must be re-evaluated prior to expiration of period of performance	External	U.S. Navy NFESC	Theresa Rojas, Program QA/QC Manager AGVIQ-CH2M HILL	Karen Koenreich, Laboratory RAM GCAL	Karen Koenreich, Laboratory RAM GCAL	Camden Robinson, Project Chemist AGVIQ-CH2M HILL

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SAP Worksheet #32—Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of CA Response Documentation	Individual(s) Receiving CA Response	Timeframe for Response
Field Performance Audit	Field Performance Audit Checklist	Field Team Tom Beisel, PM Theresa Rojas, Program QA/QC Manager AGVIQ-CH2M HILL	Within 1 day of audit	Verbal and CA Form	Andrew O'Connor, FTL AGVIQ-CH2M HILL	Within 1 day of receipt of CA Form
Safe Work Observation	Safe Work Observation Form	Field Team Andrew O'Connor, FTL Tom Beisel, PM AGVIQ-CH2M HILL	Immediately (person involved or observed person). Following day (field team). Within 1 week if worthy of elevation (H&S officer)	On Safe Work Observation Form	Andrew O'Connor, FTL, and individual being observed, and Tom Beisel, PM, and if elevated to Mike Goldman, H&S officer. AGVIQ-CH2M HILL	Corrected in the field immediately, and within 1 week if elevated.
Offsite Laboratory Technical Systems Audit	Written Audit Report	Karen Koenreich, Laboratory RAM GCAL	Within 2 months of audit	Memorandum	NFESC Auditor, TBD	Within 2 months of receipt of initial notification

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SAP Worksheet #32-1—Corrective Action Form

Person initiating CA _____ Date _____

Description of problem and when identified: _____

Cause of problem, if known or suspected: _____

Sequence of CA: (including date implemented, action planned and personnel/data affected) _

CA implemented by: _____ Date: _____

CA initially approved by: _____ Date: _____

Follow-up date: _____

Final CA approved by: _____ Date: _____

Information copies to:

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SAP Worksheet #32-2—Field Performance Audit Checklist

Project Responsibilities

Project No.:

Date:

Project Location:

Signature:

Team Members:

- | | | |
|-----|----|---|
| Yes | No | 1) Is the approved work plan being followed?
Comments: |
| Yes | No | 2) Was a briefing held for project participants?
Comments: |
| Yes | No | 3) Were additional instructions given to project participants?
Comments: |

Sample Collection:

- | | | |
|-----|----|---|
| Yes | No | 1) Is there a written list of sampling locations and descriptions?
Comments: |
| Yes | No | 2) Are samples collected as stated in the Master SOPs?
Comments: |
| Yes | No | 3) Are samples collected in the type of containers specified in the work plan?
Comments: |
| Yes | No | 4) Are samples preserved as specified in the work plan?
Comments: |

- | | | |
|-----|----|---|
| Yes | No | 5) Are the number, frequency, and type of samples collected as specified in the work plan?
Comments: |
| Yes | No | 6) Are QA checks performed as specified in the work plan?
Comments: |
| Yes | No | 7) Are photographs taken and documented?
Comments: |

Document Control:

- | | | |
|-----|----|--|
| Yes | No | 1) Have any accountable documents been lost?
Comments: |
| Yes | No | 2) Have any accountable documents been voided?
Comments: |
| Yes | No | 3) Have any accountable documents been disposed of?
Comments: |
| Yes | No | 4) Are the samples identified with sample tags?
Comments: |
| Yes | No | 5) Are blank and duplicate samples properly identified?
Comments: |
| Yes | No | 6) Are samples listed on a chain-of-custody record?
Comments: |
| Yes | No | 7) Is chain-of-custody documented and maintained?
Comments: |

SAP Worksheet #32-3—Safe Work Observation Form

Project:		Observer:		Date:
Position/Title of worker observed:		Background Information/comments:		
Task/Observation Observed:				
<ul style="list-style-type: none"> ❖ Identify and reinforce safe work practices/behaviors ❖ Identify and improve on at-risk practices/acts ❖ Identify and improve on practices, conditions, controls, and compliance that eliminate or reduce hazards ❖ Proactive PM support facilitates eliminating/reducing hazards (do you have what you need?) ❖ Positive, corrective, cooperative, collaborative feedback/recommendations 				
Actions & Behaviors	Safe	At-Risk	Observations/Comments	
Current and accurate Pre-Task Planning/Briefing (Project Safety Plan, Safety Task Analysis Card, Activity Hazard Analysis, Pre-Task Safety Plan, tailgate briefing, etc., as needed)			Positive Observations/Safe Work Practices:	
Properly trained/qualified/experienced				
Tools/equipment available and adequate				
Proper use of tools			Questionable Activity/Unsafe Condition Observed:	
Barricades/work zone control				
Housekeeping				
Communication				
Work Approach/Habits				
Attitude				
Focus/attentiveness			Observer's CAs/Comments:	
Pace				
Uncomfortable/unsafe position				
Inconvenient/unsafe location				
Position/Line of fire				
Apparel (hair, loose clothing, jewelry)				
Repetitive motion			Observed Worker's CAs/Comments:	
Other...				

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SAP Worksheet #33—QA Management Reports Table

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Field Audit Report	One during sampling activities	Submitted with report in which data is analyzed and presented	Tom Beisel, PM AGVIQ-CH2M HILL	AGVIQ-CH2M HILL Regional Health, Safety, Environment, and Quality Manager, Included in project files
Field Progress Reports (during construction activities, only)	Daily	Weekly or daily reporting will be submitted the following Monday during construction activities.	Site Superintendent	Reports will be included as an attachment to the Construction Completion Report. Construction Completion Report distribution will include the Navy and EPA
QA Management Report/Technical Memorandum	Once results are received from data validator	TBD	AGVIQ-CH2M HILL	AGVIQ-CH2M HILL, EPA Region II, PREQB, NAVFAC SE

The following will be addressed in the QA/QC section of QA Management Report/Technical Memorandum:

- Summary of project QA/QC programs and trainings
- Conformance of project activities to SAP requirements and procedures
- Status of project and schedule delays
- Deviations from approved SAP and approved amendments to SAP
- Description and findings of audits
- Results of data review activities in terms of amount of usable data generated (results of the Chemist's QC check on data prior to loading into AGVIQ-CH2M HILL's database)
- Required corrective actions and effectiveness of corrective action implementation
- Data usability assessments in terms of accuracy, precision, representativeness, completeness, comparability and sensitivity
- Limitations on use of measurement data generated

The report will also include data quality concerns:

- Narrative and timelines of project activities summary of project quality objective (PQO) development
- Reconciliation of project data with PQOs
- Summary of major problems encountered and their resolution
- Data summary, including tables, charts, graphs, with appropriate sample identification or station location numbers, concentration units, percent solids (not applicable), and data quality flags
- Conclusions and recommendations

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SAP Worksheet #34—Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification
Field Notebooks	Field notebooks will be reviewed internally and placed into the project file for archival at project closeout.	Internal	Andrew O'Conor/AGVIQ-CH2M HILL
Chains of Custody and Shipping Forms	Chain-of-custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain-of-custody will be initialed by the reviewer, a copy of the chain-of-custody retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	Internal	Andrew O'Conor/AGVIQ-CH2M HILL
Sample Condition upon Receipt	Any discrepancies, missing, or broken containers will be communicated to the project chemist or designee in the form of laboratory logins.	Internal	Project Chemist or designee: Camden Robinson/AGVIQ-CH2M HILL
Sample Chronology	Holding times from collection to extraction or analysis and from extraction to analysis will be considered by the data validator during the data validation process.	External and Internal	Third Part Data Validator: Mike Stewart/eDATApro Project Chemist: Camden Robinson/ AGVIQ-CH2M HILL
Documentation of Laboratory Method Deviations	Laboratory method deviations will be discussed and approved by the project chemist. Documentation will be incorporated into the case narrative which becomes part of the final hardcopy data package.	Internal	Project Chemist: Camden Robinson/AGVIQ-CH2M HILL
Electronic Data Deliverables	Electronic data deliverables will be compared against hardcopy laboratory results.	Internal	Project Data Coordinator: Kama White/AGVIQ-CH2M HILL
Case Narrative	Case narratives will be reviewed by the data validator during the data validation process.	External and Internal	Third Party Data Validator: Mike Stewart/eDATApro Project Chemist: Camden Robinson/AGVIQ-CH2M HILL

SAP Worksheet #34—Verification (Step I) Process Table (continued)

Verification Input	Description	Internal/ External	Responsible for Verification
Laboratory Data	<p>All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal.</p> <p>All received data packages will be verified internally by the project chemist. 10% of data packages received will be verified externally by the third party validator.</p>	Internal and External	<p>Respective Laboratory QA Officer</p> <p>Third Party Data Validator: Mike Stewart/eDATApro</p> <p>Project Chemist: Camden Robinson/AGVIQ-CH2M HILL</p>
Audit Reports	<p>Upon report completion, a copy of all audit reports will be placed in the site file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the QA site file. Periodically, and at the completion of site work, site file audit reports and corrective action forms will be reviewed internally to ensure that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the site manager will be notified to ensure action is taken.</p>	Internal	<p>Project Manager: Tom Beisel/AGVIQ-CH2M HILL</p> <p>QC Manager: Eric Burrell/AGVIQ-CH2M HILL</p> <p>Program QA/QC Manager: Theresa Rojas/AGVIQ-CH2M HILL</p>
Corrective Action Reports	<p>Corrective action reports will be reviewed by the project chemist or project manager and placed into the project file for archival at project closeout.</p>	Internal	<p>Project Chemist: Camden Robinson/AGVIQ-CH2M HILL</p> <p>Project Manager: Tom Beisel/AGVIQ-CH2M HILL</p>

SAP Worksheet #35—Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation
IIb	Onsite Screening	Ensure that all field data meet SAP requirements for completeness and accuracy based on the field calibration records.	Andrew O'Connor/AGVIQ-CH2M HILL
IIa	SOPs	Ensure that all sampling and analytical SOPs were followed.	Andrew O'Connor/AGVIQ-CH2M HILL Karen Koenreich/GCAL
IIa	Method QC Results	Ensure that all required QC samples were run and met method and/or project required limits.	Mike Stewart/eDATapro Camden Robinson/AGVIQ-CH2M HILL
IIb	SAP QC Sample Results	Ensure that all required SAP QC samples were run and met required limits.	Camden Robinson/AGVIQ-CH2M HILL Mike Stewart/eDATapro
IIb	QLs	Ensure all sample results met the project quantification limit specified in the SAP.	Camden Robinson/AGVIQ-CH2M HILL
IIa	Raw Data	Ten percent review of raw data to confirm laboratory calculations	Mike Stewart/eDATapro
IIa	Raw Data	Review all raw data to confirm laboratory calculations.	Camden Robinson/AGVIQ-CH2M HILL
<p>Notes:</p> <p>IIa = compliance with methods, procedures, and contracts IIb = comparison with measurement performance criteria in the SAP</p>			

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SAP Worksheet #36—Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa	Aqueous	VOCs; MEE; Dissolved Metals – Iron, Manganese; General Chemistry Parameters – TOC; Sulfate; Sulfide, Alkalinity, Dehalococoides Ethenogenes	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers that may be used are those presented in EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008) and Inorganic Data Review (January 2010) using guidance of DOD QSM - Version 3.0 ^a Final April 22, 2010 (based on NELAC Voted Version 5 – June 2003).	Mike Stewart/ eDATApro Camden Robinson/ AGVIQ-CH2M HILL
IIa	Aqueous	VOCs; Dissolved Iron; General Chemistry Parameters – TOC; Sulfate; Nitrate; Sulfide; COD; Alkalinity	Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers that may be used are those presented in EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (June 2008) and Inorganic Data Review (January 2010) using guidance of DOD QSM – Version 3.0 ^a Final April 22, 2010(based on NELAC Voted Version 5 – June 2003).	Mike Stewart/ eDATApro Camden Robinson/ AGVIQ-CH2M HILL
IIa	Aqueous	VOCs; MEE; Dissolved Metals – Iron, Manganese; General Chemistry Parameters – TOC; Sulfate; Sulfide, Alkalinity, Dehalococoides Ethenogenes	Data will be reviewed against the analytical methods for outstanding QA/QC issues and anomalies by the laboratory. Issues will be summarized in the case narrative. AGVIQ-CH2M HILL chemist and PM will review the analytical results and case narrative before the data is loaded to ensure no major problems exist.	Karen Koenreich/GCAL Camden Robinson/ AGVIQ-CH2M HILL Tom Beisel/ AGVIQ-CH2M HILL
IIa	Aqueous	VOCs; Dissolved Iron; General Chemistry Parameters – TOC; Sulfate; Nitrate; Sulfide; COD; Alkalinity	Data will be reviewed against the analytical methods for outstanding QA/QC issues and anomalies by the laboratory. Issues will be summarized in the case narrative. AGVIQ-CH2M HILL chemist and PM will review the analytical results and case narrative before the data is loaded to ensure no major problems exist.	Karen Koenreich/GCAL Camden Robinson/ AGVIQ-CH2M HILL Tom Beisel/ AGVIQ-CH2M HILL
IIb	Aqueous	VOCs; MEE; Dissolved Metals – Iron, Manganese; General Chemistry Parameters – TOC; Sulfate; Sulfide, Alkalinity, Dehalococoides Ethenogenes	Results will be compared to Project Action Limits in Worksheet #15.	Camden Robinson/ AGVIQ-CH2M HILL Tom Beisel/ AGVIQ-CH2M HILL

SAP Worksheet #36—Analytical Data Validation (Steps IIa and IIb) Summary Table (continued)

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIb	Aqueous	VOCs; Dissolved Iron; General Chemistry Parameters – TOC; Sulfate; Nitrate; Sulfide; COD; Alkalinity	Results will be compared to Project Action Limits in Worksheet #15.	Camden Robinson/ AGVIQ-CH2M HILL Tom Beisel/ AGVIQ-CH2M HILL

Notes:

^a In order to stay consistent with historical data and to meet the requirements of the regulatory documents, this SAP is using guidance of DOD QSM - Version 3.0.

IIa = compliance with methods, procedures, and contracts

IIb = comparison with measurement performance criteria in the SAP

Appendix D presents the data validation checklists for SW-846. A Level IV data package is required for this project, and data package deliverables are presented in Appendix D. For a Level IV data package, chromatograms are included before and after each of the manual integrations. Manual integrations are evaluated based on the following process:

Manual integrations are an integral part of the chromatographic analysis process and will be used only as a CA measure. Examples of instances where manual integration would be warranted include, but are not limited to, co-eluting compounds resulting in poor-peak resolution, a misidentified peak, an incorrect retention time, or a problematic baseline.

When manual integrations are used, the following procedures will be implemented for documenting the event and for consistency in performing the manual integration:

- A laboratory SOP will be followed for manual integrations. This SOP will specify: (1) when automated integrations by the instrument are likely to be unreliable; (2) what constitutes an unacceptable automated integration; (3) how the problems should be resolved by the analyst; and (4) the procedures for the analyst to follow in documenting any required manual integrations.
- Raw data records will include a complete audit trail for those manipulations, including: (1) results of both the automated and manual integrations; (2) notation of the cause and justification for performing the manual integrations; (3) date; and (4) signature or initials of person performing the manual operations.
- All manual integrations must be reviewed and approved by the section supervisor and/or the QA officer.

SAP Worksheet #37—Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following sections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision-making for the project, and describes how data quality issues will be addressed and how limitations of the use of the data will be handled.

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

- Data gaps may be present if: (1) a sample is not collected; (2) a sample is not analyzed for the requested parameters; or (3) the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis, depending on whether data can be extrapolated from adjacent sample locations, and whether the data are needed based on the results from adjacent sample locations.
- Non-detected site contaminants will be evaluated to ensure that project-required quantitation limits in Worksheet #15 were achieved. If project quantitation limits were achieved and the verification and validation steps yielded acceptable data, then the data is considered usable.
- During verification and validation steps, data may be qualified as estimated with the following qualifiers: J, UJ, B, or JB. These qualifiers represent minor QC deficiencies which will not affect the usability of the data. When major QC deficiencies are encountered, data will be qualified with an R or UR and in most cases is not considered usable for project decisions.
- For statistical comparisons non-detect values will be represented by a concentration equal to one-half the sample reporting limit. For duplicate sample results, the most conservative value will be used for project decisions.
- Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. These checks include comparison of hardcopy data and qualifiers to the electronic data deliverable. Once the data has been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately.
- Field and laboratory precision will be compared as RPD between the two results.
- Deviations from the UFP-SAP will be reviewed to assess whether corrective action is warranted and to assess impacts to achievement of project objectives.

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

- To assess whether a sufficient quantity of acceptable data are available for decision making, the data will be reconciled with measurement performance criteria following validation and review of data quality indicator.

- If significant biases are detected with laboratory QA/QC samples it will be evaluated to assess impact on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site.
- If significant deviations are noted between lab and field precision the cause will be further evaluated to assess impact on decision making.

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

The following will be prepared by AGVIQ-CH2M HILL and presented to and submitted to the Tier I Partnering Team for review and decisions on the path forward for the site.

- Data tables will be produced to reflect detected and non-detected site COCs. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation.
- Graphical representations will be produced to reflect increasing and/or decreasing concentrations of COCs and geochemical parameters.
- Maps will be produced to reflect increasing and/or decreasing areas of soil contamination.
- A data quality evaluation considering all of the above will be provided as part of presentations to the Tier I Partnering Team, followed by the technical memorandum prepared to assess remedy effectiveness. The technical memorandum will identify any data usability limitations and make recommendations for corrective action if necessary.

Data Quality Evaluation

The Project Chemist or designee will perform the DQE. The DQE process is used to assess the effect of the overall analytical process on the usability of the data. The two major categories of data evaluation are laboratory performance and matrix interferences. Evaluation of laboratory performance is a check for compliance with the method requirements. It is a straight-forward examination: either the laboratory did, or did not, analyze the samples within the limits of the analytical method. Evaluation of the matrix interferences is more subtle and involves analysis of several results, including surrogate spike recoveries, matrix spike recoveries, and duplicate sample results. The project team will evaluate the data validation results. This evaluation will assess how the data, as qualified by the data validation, can be used on the project.

Once each of the data packages has been validated, and the data validation worksheets completed, then the entire data set will be evaluated for overall trends in data quality and usability.

Information summarized as part of the DQE may include chemical compound frequencies of detection, dilution factors that might affect data usability, and patterns of target compound distribution. The data set also will be evaluated to identify potential data limitations or uncertainties in the laboratory.

Reconciliation with Data Quality Objectives

The final activity of the DQE process is to assess whether the data meet the planned DQOs for the project. The final results, as adjusted for the findings of any data validation and data evaluation, will be checked against the DQOs, and an assessment will be made as to whether the data are of sufficient quality to support the DQOs. The decision as to data sufficiency may be affected by the

overall precision, accuracy, and completeness of the data as demonstrated by the data validation process. The main project objective should be met assuming the 90 percent completeness goal is obtained after all of the data have undergone sufficient data validation. If the data, after validation and evaluation, are sufficient to achieve project objectives, the data quality and project managers will release the data and work may proceed.

Identify the personnel responsible for performing the usability assessment:

The AGVIQ-CH2M HILL team, including the PM, Project Chemist, and Senior ERD Technologist, will review the data and compile a presentation for the Partnering Team. The Tier I Partnering Team as a whole will assess the usability of the data.

Usability Assessment Documentation

All the results will be assembled and statistically reported for an overall quality assessment. AGVIQ-CH2M HILL will prepare a report that will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field and lab duplicates. The report will also include discussions regarding the precision, accuracy, representativeness, comparability, and completeness. The report will also include data tables to reflect detected and non-detected site contaminants and geochemical parameters. Data qualifiers will be reflected in the tables and discussed in the DQE. Figures will be produced representing contaminant concentrations.

Precision

Laboratory precision is measured by the variability associated with duplicate (two) or replicate (more than two) analyses. One type of sample that can be used to assess laboratory precision is the LCS. Multiple LCS analyses over the duration of the project can be used to evaluate the overall laboratory precision for the project. In this case, the comparison is not between a sample and a duplicate sample analyzed in the same batch, but between LCSs analyzed in multiple batches.

Total precision is the measurement of the variability associated with the entire sampling and analytical process. The required level of precision for each method, matrix, and analyte are provided in Worksheet #15. The level of precision is determined by analysis of duplicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate and MSD samples will be analyzed to assess field and laboratory precision at a frequency described in Worksheet #20. For duplicate sample results, the precision is evaluated using the RPD. For replicate results, the precision is measured using the RSD. The formulas for the calculation of RPD and RSD are provided below.

If calculated from duplicate measurements:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2) / 2} \tag{1}$$

Where:

- RPD = relative percent difference
- C₁ = larger of the two observed values
- C₂ = smaller of the two observed values

If calculated from three or more replicates, use RSD rather than RPD:

$$RSD = (s / \bar{y}) \times 100\% \quad (2)$$

Where:

RSD = relative standard deviation

s = standard deviation

\bar{y} = mean of replicate analyses

Standard deviation, s, is defined as follows:

$$S = \sqrt{\sum_{i=1}^n \frac{(y_i - \bar{y})^2}{n - 1}} \quad (3)$$

Where:

S = standard deviation

y_i = measured value of the i^{th} replicate

\bar{y} = mean of replicate analyses

n = number of replicates

Accuracy

Accuracy reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. Analytical accuracy is measured by comparing the percent recovery (%R) of analytes spiked into an LCS to a control limit. For many methods of organic compound analysis, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The formula for calculation of accuracy is presented below as %R from pure and sample matrices. Accuracy requirements are listed for each method, matrix, and analyte in Worksheet #28.

For measurements where MS are used:

$$\%R = 100\% \times \left[\frac{S - U}{C_{sa}} \right] \quad (4)$$

Where:

%R = percent recovery

S = measured concentration in spiked aliquot

U = measured concentration in unspiked aliquot

C_{sa} = actual concentration of spike added

For situations where a standard reference material (SRM) is used instead of or in addition to MS:

$$\%R = 100\% \times \left[\frac{C_m}{C_{sm}} \right] \quad (5)$$

Where:

$\%R$ = percent recovery

C_m = measured concentration of SRM

C_{sm} = actual concentration of SRM

Representativeness

Representativeness is a qualitative term that refers to the degree in which data accurately and precisely depicts the characteristics of a population, whether referring to the distribution of contaminant within a sample, a sample within a matrix, or the distribution of a contaminant at a site. Representativeness is determined by appropriate program design, with consideration of elements, such as proper well locations, drilling and installation procedures, operations process locations, and sampling locations. Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Assessment of representativeness will be achieved through use of the standard field, sampling, and analytical procedures. Decisions regarding sample/well/boring locations process and numbers and the statistical sampling design are documented in Worksheets #10, #11, and #17.

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms will support the assessment of comparability. Historical comparability will be achieved through consistent use of methods and documentation procedures throughout the project. Assessment of comparability is primarily subjective and results should be interpreted by experienced environmental professionals with a clear knowledge of the PQOs and project decisions.

Completeness

Completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. It is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (for example, by site) as set out in the PQOs. Valid data are data that are usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed. Completeness should not be determined only on the basis of laboratory data qualifiers. The goal for completeness is 95 percent for aqueous samples and 90 percent for soil samples.

Defined as follows for all measurements:

$$\%C = 100\% \times \left[\frac{V}{T} \right] \quad (6)$$

Where:

$\%C$ = percent completeness

V = number of measurements judged valid

T = total number of measurements

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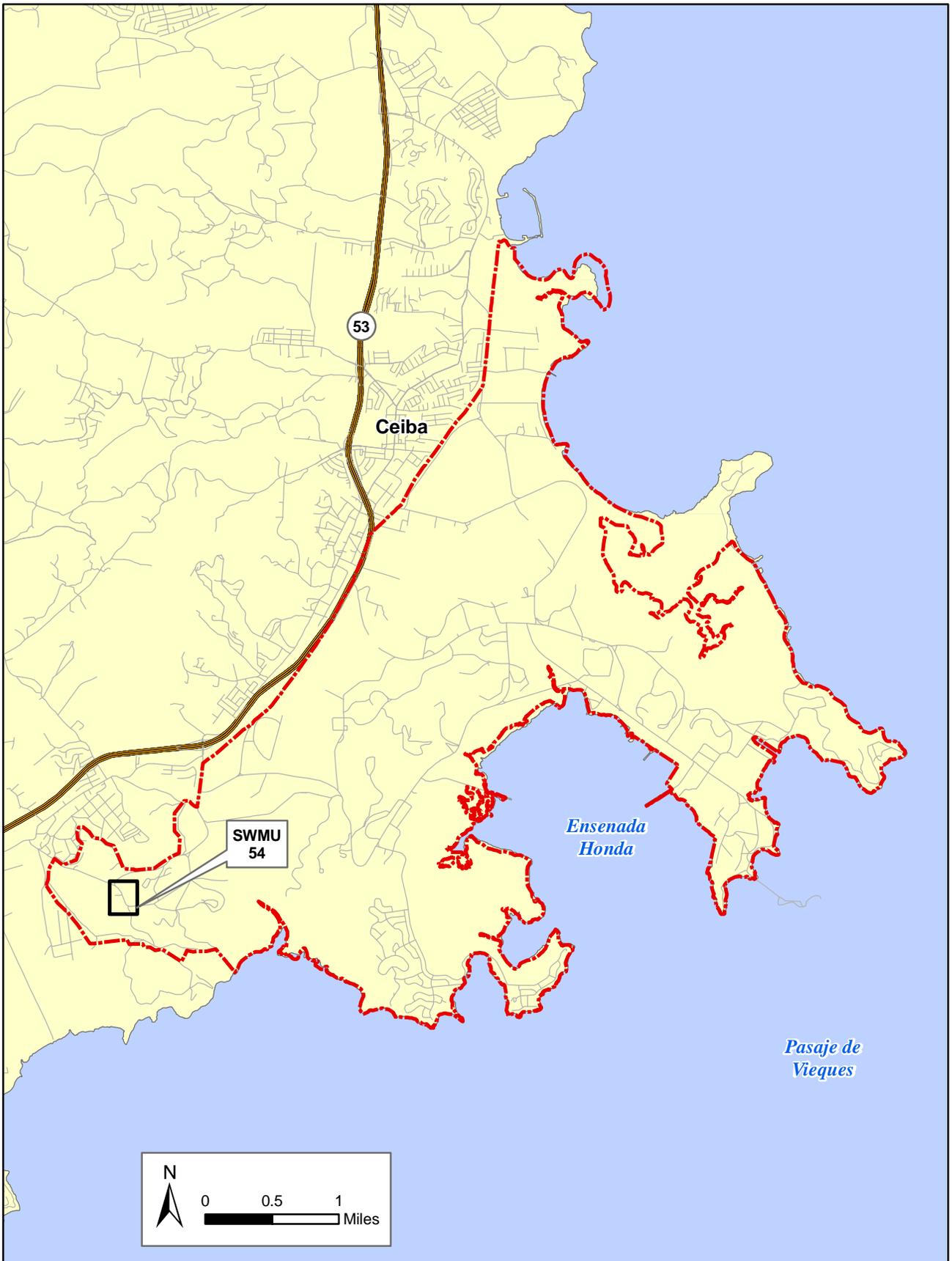
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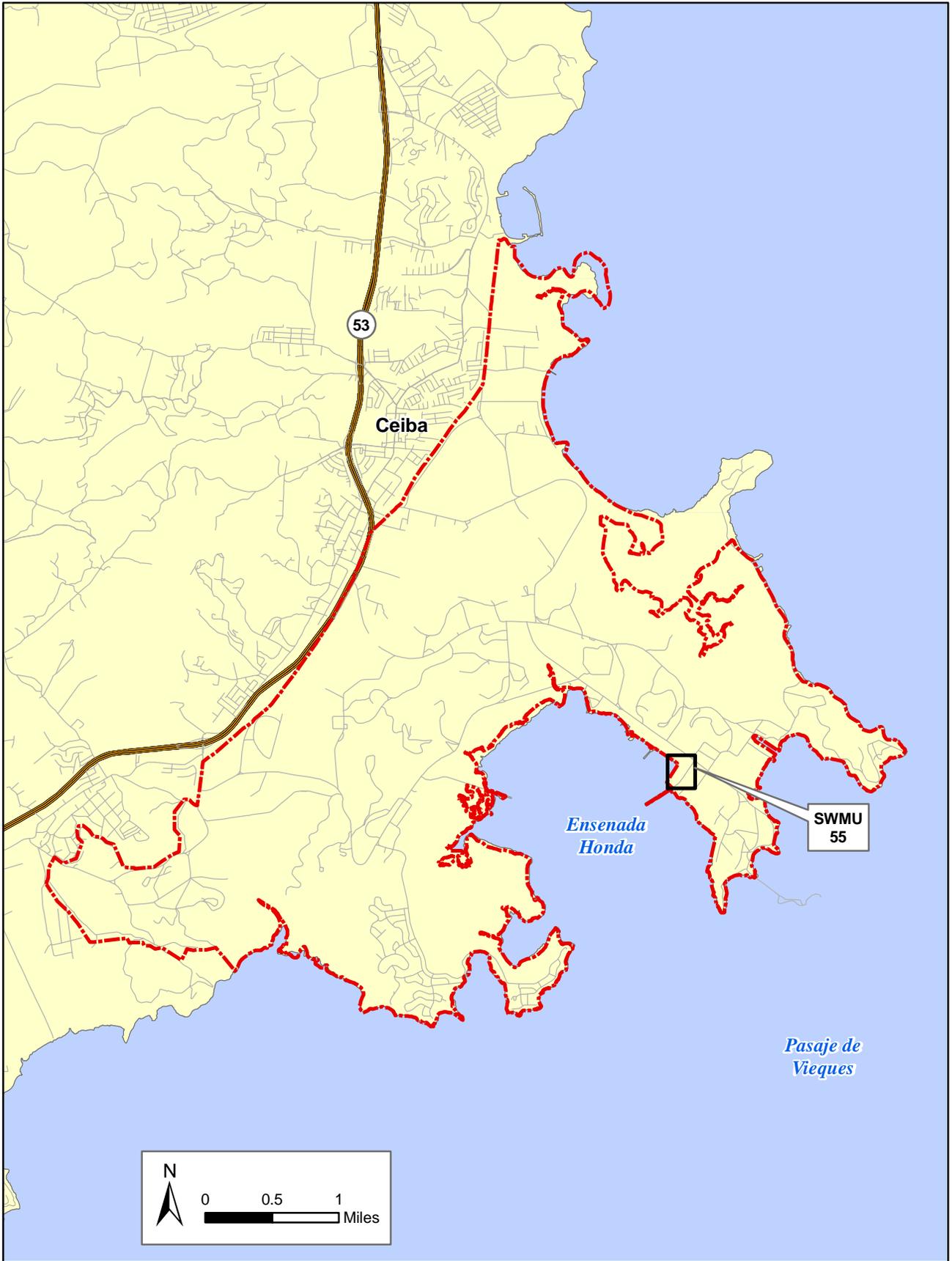
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APPENDIX A
Figures



- Road
- Expressway
- Naval Station Roosevelt Roads Boundary

FIGURE 1
 SWMU 54 Location
Naval Station Roosevelt Roads, Puerto Rico



- Road
- Expressway
- ▭ Naval Station Roosevelt Roads Boundary

FIGURE 2
 SWMU 55 Location
Naval Station Roosevelt Roads, Puerto Rico



- Monitoring Well Screened Primarily Less than 15 ft bgs
- Monitoring Well Screened Primarily Greater than 15 ft bgs
- ▲ Injection Well Screened 17-27 ft bgs
- Air Sparge Injection Well
- Former Structure

Originated By: Amanda Struse *Amanda Struse*
 Checked By: Shruti Shah *Shruti Shah*
 Checked By: Alicia Nobles *Alicia Nobles*

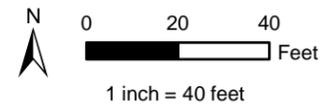
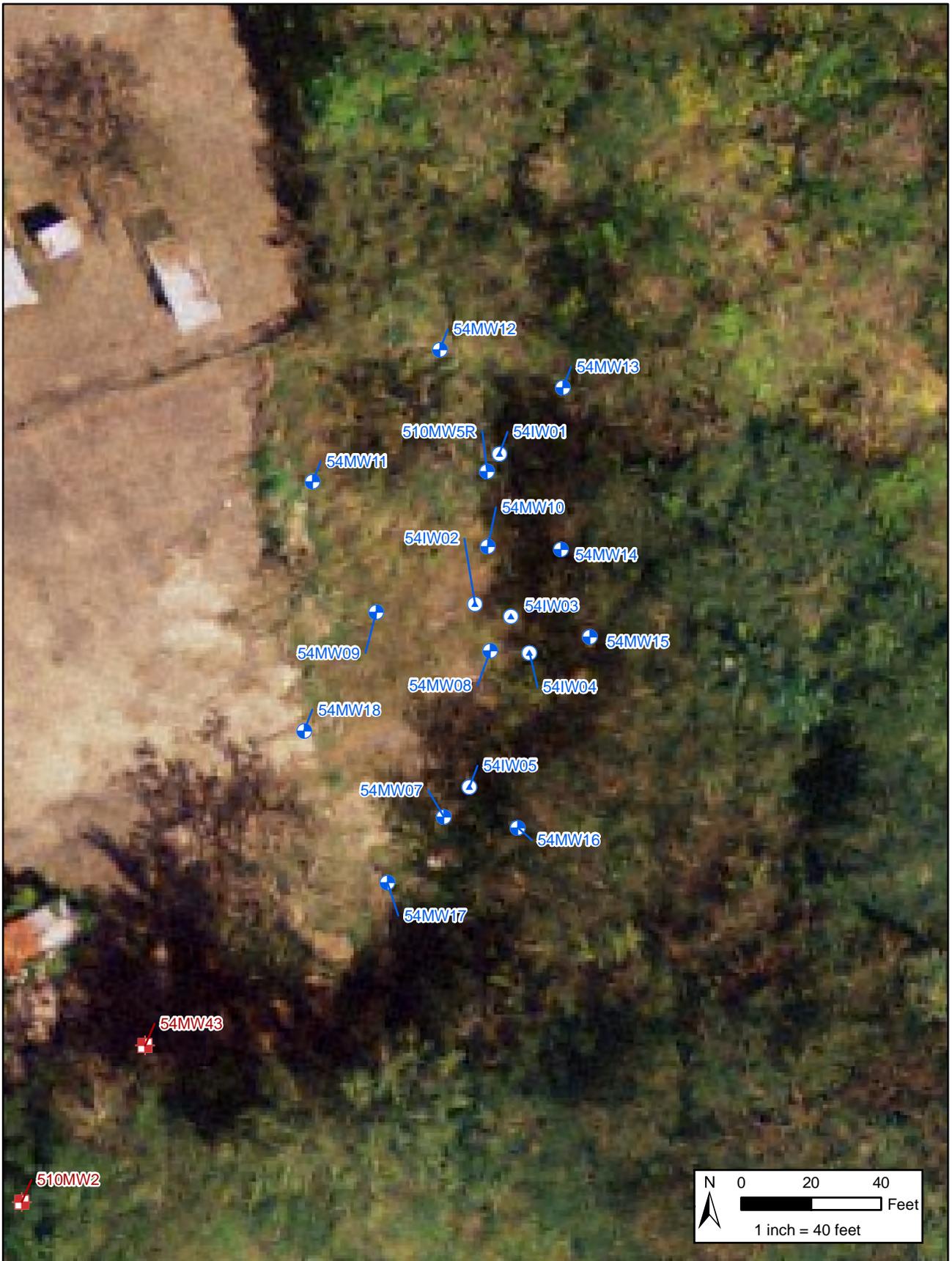


FIGURE 1-2
 Site Layout
 SWMU 54
 Naval Activity Puerto Rico



Note: CAO for TCE = 22 µg/L

- Monitoring Well Screened
Primarily Less than 15 ft bgs
- ⊕ Monitoring Well Screened
Primarily 15-25 ft bgs
- ⊕ Injection Well Screened
17-27 ft bgs

Originated By: Amanda Struse *Amanda Struse*
 Checked By: Alicia Nobles *Alicia Nobles*

FIGURE 2-1
 TCE Plume Well Layout
 SWMU 54
 Naval Activity Puerto Rico



- Monitoring Well Screened Primarily Less than 25 ft bgs
- Injection Well Screened Primarily Less than 25 ft bgs
- Monitoring Well Screened Primarily Greater than 25 ft bgs
- Injection Well Screened Primarily Greater than 25 ft bgs
- Monitoring Well
- Injection Well
- SWWU 55 Boundary

Originated By: Amanda Struse *Amanda Struse*
 Checked By: Kimberley Stokes *Kimberley Stokes*

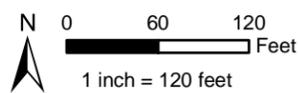


FIGURE 1-2
 Site Layout
 SWMU 55
 Naval Activity Puerto Rico

Standard Operating Procedures

Standard Operating Procedures (SOPs)

The following Standard Operating Procedures (SOPs) will be used, as applicable, during the pilot studies at SWMU 54 and 55 sampling event to ensure that consistent methods are used and that defensible data are collected. Any deviations from these SOPs will be approved by the Navy and properly documented.

Documentation

Injections (One Permanent Well and One DPT)

Drilling Procedures

Purpose and Applicability

This procedure conforms to the applicable EPA Quality Assurance Requirements. The procedure describes the method for installing monitoring wells.

Definitions

DPT: Direct Push Technology
HSA: Hollow-Stem Auger

Procedure

All drilling activities shall conform to state and local regulations and shall be supervised by a professional geologist, engineer, or hydrogeologist. The driller shall obtain all necessary permits, applications, and other documents required by state and local authorities.

The location of all borings shall be coordinated with drilling subcontractor point of contact (POC) or equivalent before drilling commences and cleared by a utility locating company. When drilling boreholes through more than one water bearing zone or aquifer, double cased wells will be constructed to prevent cross-connection or cross-contamination of the zones or aquifers (EPA Region 4, May 1996).

Prior to each monitoring well installation, the drilling rig, equipment, and tools should be decontaminated according to the decontamination procedures described in this document. The drill rig should not leak any fluids, because the fluids could enter the borehole or contaminate equipment placed in the hole. The use of rags or absorbent materials to absorb leaking fluids is unacceptable.

Lubricants with constituents that are toxic or that increase, decrease, or mask the target chemical species of the investigation shall not be permitted. The driller shall be prepared to provide chemical analyses of all lubricants proposed for downhole use. Chemical detection limits shall be equivalent to those used in analyzing project groundwater samples.

A log of drilling activities shall be kept in a bound field notebook. Information in the log book shall include location, time on site, personnel present, equipment present, down time, materials used, samples collected, measurements taken, and any other observations or information that would be necessary to reconstruct field activities at a later date.

Direct Push Technology and Geoprobe™

The direct push technology (DPT) and Geoprobe units are rugged, lightweight hydraulic and/or hammer driven systems utilized to advance soil borings with minimal lithological disturbance, minimal IDW production, and minimal time for evaluation of contaminant extent.

Drilling and sampling procedures for DPT and Geoprobe are similar.

- DPT and Geoprobe units are generally operated from a station mounted at the rear of a truck, bobcat, or four-wheeler-mounted unit. The probing unit is hydraulically and/or hammer driven via a power takeoff pump mounted directly to the truck's transmission.
- Soil samples may be obtained by using a wide variety of devices. Sampling devices range from 2-inch O.D. (outside diameter) tubes to sampling systems that allow for the collection of continuous soil samples.
- The samplers are threaded onto the leading edge of the DPT drive rods and advanced to depth using the direct push systems.
- Soil samples are retrieved by retracting the probe rod and sampler to the surface and disassembling the sampler.
- Samples are obtained in sleeves made of brass, stainless steel, Teflon® or acetate. The sleeves are removed from the sampler, containerized, and transported to the laboratory for analysis.

Hollow-Stem Auger Drilling Methods

This type of drilling method consists of using a hollow, steel stem or shaft auger with a continuous, spiraled steel flight welded to its exterior and an auger bit connected to its end. The hollow-stem auger transports cuttings to the surface when rotated. This method is best suited to soils that have a tendency to collapse when disturbed. A monitoring well can be installed inside a hollow-stem auger with little or no concern for the caving potential of the soils and/or water table. However, retracting an auger in caving sand conditions while installing a monitoring well can be extremely difficult or impossible, especially since the auger has to be extracted without being rotated.

If caving sand conditions exist during monitoring well installation, a drilling rig must be used that has enough power to extract the auger from the borehole without having to rotate it. A bottom plug, trap door, or pilot bit assembly can be fastened to the bottom of the auger to keep out the soils and water that have a tendency clog the bottom of the auger during drilling. Potable water (analyzed for contaminants of concern) may be poured into the auger (where applicable) to equalize pressure so that the inflow of formation materials and water will be held to a minimum when the bottom plug is released. A watertight center plug is not acceptable because it creates suction when extracted from the auger. This suction forces or

pulls cuttings and formation materials into the auger, 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 auger is removed by either washing out the plug using a side discharge rotary bit, or by augering out the plug with a solid-stem auger bit sized to fit inside the hollow-stem auger. Prior to drilling operations, the type of bottom plug, trap door, or pilot bit assembly proposed for the drilling activity should be approved by a senior field geologist and AFCEE. 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 (EPA Region 4, May 1996).

Drilling and sampling procedures for HSA are as follows:

1. The HSA is advanced and sampled either at discrete intervals, continuously (18-inch intervals), or with "CME-type" 5-foot long continuous sampler.
2. The HSA sampler consists of a split-spoon sampling device, which is a thick-walled, steel tube split lengthwise. A cutting shoe is attached to the lower end of the tube; the upper end is connected to the drill rods. The split-spoon sampler is lowered into the hole on the bottom of the drill rods, and into the soil ahead of the auger bit.
3. The density of the sampled material is obtained by counting the blows per foot (blow count). The split spoon sampler is driven into the soil by a 140-pound hammer which falls at 30 inches intervals per blow.
4. Soil samples are retrieved by retracting the split-spoon rod and split-spoon to the surface. Brass or stainless steel rings should be used in the split-spoon sampler when sampling for VOCs.
5. Groundwater samples may be obtained utilizing either inert nylaflow tubing and sampling syringe, stainless steel bailer, a jiggle tube type of pump, or a peristaltic pump. Groundwater samples may also be taken via a temporary or permanent piezometer or monitoring well, where sampling can be accomplished at any time using bailers, jiggle tubes, pumps, or other methods.

Monitoring Well Installation

Purpose and Applicability

This procedure conforms to the EPA Quality Assurance Requirements and describes the method for installing permanent monitoring wells.

Definitions

None.

Procedure

The methods for installing permanent monitoring wells are described below.

Permanent Monitoring Wells

After adequate soil and groundwater samples have been collected to characterize the soil boring lithologically, hydrologically, and chemically, the well screen and casing are installed to depth. A minimum 2-inch annular space is required between the casing and the borehole wall. The well casing, screen, sediment trap, and end cap are assembled and installed so as to prevent damage to the sections and joints. No glue, solvents, or pipe dope should be used on casing threads to secure casing joints.

Well Casing and Screen Assembly

The casing for the monitoring wells is new, unused, threaded Schedule 40 PVC pipe (such as pipe manufactured by Brainard-Kilman). Joints are flush-threaded and assembled with Teflon® tape. O-rings are removed prior to assembly; however, if the O-rings are made of Teflon®, they can be used in the well assembly to insure a tight fit of casing joints.

The well screens are new, unused, factory-made, machine-slotted Schedule 40 PVC pipe. Screens will be 10 feet in length and will be placed at the bottom of the well. Each well screen is sealed by a threaded end cap. The screen has flush-threaded joints compatible with the well casing. Threaded joints are secured with Teflon® tape to insure a tight fit of the casing joints.

Before the well casing and screen 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 top of the casing has a temporary cap during installation of the annulus materials.

Filter Pack Installation

After the casing and screen assemblies are set at the appropriate depth, the sand filter pack is inserted. The sand filter pack consists of a thoroughly washed, sound, durable, siliceous material containing less than 5 percent silt or clay (commercially available 20/30-grain size or equivalent). No organic material, anhydrite, gypsum, mica, or calcareous material is allowed. The minimum specific gravity of the sand pack material is 2.5. No water is used unless approved. The filter pack is installed around the well screen (preferably using a tremie) in approximate 2-foot lifts to prevent bridging. The depth to the top of the sand filter pack is measured periodically using a weighted measuring tape.

Transition Seal

At least 3 feet of bentonite transition seal is placed above the sand filter pack. The bentonite seal is in pellet form. (**Note:** If wells are deeper than 15 feet, then seal and grout must be tremmied down borehole). Depths to the bottom and top of the bentonite seal are measured and documented to ensure that the transition seal meets design requirements. The bentonite is allowed to hydrate for at least 1 hour prior to beginning emplacement of the cement-bentonite grout. Potable water may be added to the borehole to hydrate well seals placed above the water table surface.

Annular Grout Seal

The grout seal is Portland cement conforming to ASTM C-150, Type 1. The maximum allowable water content of the grout mix is 7 gallons per 94-pound sack of cement. The maximum amount of bentonite allowable in the grout mix is 2.7 pounds per 94-pound sack of cement. Bentonite is either mixed into the water prior to adding the cement or it is mixed into the cement powder prior to adding water. The grout is mixed thoroughly before being placed in the borehole.

Surface Completion of Well

For wells located in paved areas, all concrete and asphalt at each boring location will be removed to create a 2-foot-square opening in the pavement (opening needs to be large enough to accommodate drilling activities and monitoring well activities subsequent to drilling). A concrete saw will be used to cut the opening in the concrete or asphalt. Jagged-edged openings will not be permitted. A jackhammer or similar tool may be required to remove the concrete pavement after cutting through the pavement with the concrete saw. The well head will be completed either by constructing a flush-mount cover consisting of a concrete slab at least 4 inches thick in the 2-foot-square opening or by a lockable steel encasement on stick-up wells. Construction of the pads and guard posts shall not begin for a minimum of 24 hours after well completion to allow the grout to cure.

The well cap will be a watertight cap or cover made specifically for the PVC well casings. The cap will be lockable and include a brass padlock. Wells will be keyed alike to match the facility's standard monitoring well lock.

Flush Mount

On flush-mounted completions, an 8-inch-diameter vault will be placed 0.25 inch above the existing pavement surface, with the concrete surface sloping smoothly from the vault surface to the existing pavement surface. The vault will be centered in the 2-foot-square pavement opening, with each vault having bolt-on traffic-bearing iron covers. The slab will be reinforced with four 20-inch-long steel reinforcing rods (#3 minimum size) placed uniformly around the vault within the concrete slab. The concrete surface will be finished smoothly, and a metal survey marker will be embedded in the fresh concrete.

Above Ground Finish

On stick-up wells, a protective outer casing with a hinged lid will be installed and centered on each monitoring well casing. At least one weep hole shall be drilled near the base of the protective cover to allow rainwater to drain out. Concrete filled guard posts (2 to 4 per well location), consisting of 3-inch diameter Schedule 40 steel, shall surround each well to protect it. The posts shall be a minimum of 5 feet in length and shall be installed to a minimum depth of 2 feet bgs in a concrete footing and extend a minimum of 3 feet above ground

surface. The protective casing and guard posts shall be painted with a rust-inhibiting paint and an acceptable color.

Well Development Procedures

Well development procedures should be completed within 48 hours after a well is installed. The wells should be developed by alternately pumping and surging until the water is visibly free of sediment. Development water will be contained as IDW. Development equipment is to be decontaminated as specified in Section C.13. Newly installed wells should not be developed for at least 24 hours after installation to allow sufficient time for the well materials to cure. Wells should be developed by surging, bailing, and pumping as follows:

1. Remove the well cap or cover and monitor for vapors using the instruments listed in the HSP.
2. Obtain depth to water measurements and determine the well volume.
3. Pump/bail the well as necessary to lower the water level and draw sediments from the sand pack into the well. Bail or pump until the water is relatively clear. Containerize this development water as IDW. For unproductive wells and to aid in the development process, a surge-block can be used to slowly swab the screened interval in between pumping/bailing.
4. As each well volume of water is removed, measure and record pH, temperature, specific conductance, and turbidity.
5. Bail or pump at least five well volumes of water from the well while taking field measurements (pH, temperature, specific conductance, and turbidity).

Development is considered complete if a minimum of five well volumes of water have been removed and three successive measurements of pH, temperature, specific conductance, and turbidity have remained stable (See Groundwater Sampling Section).

Cleaning and Decontamination of Sampling Equipment

Purpose and Applicability

This procedure conforms to the applicable EPA Quality Assurance Requirements and it establishes standard methodologies for cleaning and decontaminating sample containers and sampling devices. The procedure applies to all field investigations.

Definitions

DOM: Delivery Order Manager

Procedure

Sampling devices are cleaned and decontaminated before and after field use, as well as between each sample collection location. Sample containers are cleaned by the supplier before issuance to field personnel. All decontamination fluids are regarded as IDW and will be containerized and disposed of as such. Specific cleaning and decontamination materials and methods are discussed below.

Cleaning and Decontamination Materials

The following materials may be used for decontamination:

1. Trisodium phosphate or a laboratory detergent, such as Alquinox, Liquinox, or the equivalent.
2. Pesticide-grade isopropanol. The DOM must justify the use of any solvent other than pesticide-grade isopropanol for cleaning and decontamination.
3. Tap water from an acceptable municipal water treatment system.
4. Organic/Metal-free water rinse.

During cleaning and decontamination operations, the substitution of higher-grade water for tap water is permitted and does not have to be noted as a variation.

Marking and Storage

Cleaned and decontaminated equipment is bagged and wrapped in aluminum foil or plastic, depending on the size of the equipment, and the decontamination process/occurrences are recorded in the field logbook. Cleaned and decontaminated items are stored in a contaminant-free environment.

Unused field equipment, reusable or disposable sample containers, and sample tubing that have been transported to a facility or site where contamination is known or suspected to be present or which may have become contaminated during the course of the field investigation should not be replaced in storage without being cleaned and decontaminated.

Decontamination Quality Control

Source Water Blank

Collecting samples in containers provided by the laboratory and submitting them for analysis monitors the quality of tap and organic-free rinse water. At least one sample per lot of organic-free water is collected and submitted for analysis, and each tap water source used for decontamination will be sampled. When field deionizing or organic-free water units are used, QC samples are collected and analyzed more frequently. An initial sample plus subsequent weekly sampling is the minimum acceptable frequency of QC sampling. 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.

Equipment Rinsate

The effectiveness of the cleaning and decontamination procedures used in the field may be monitored by rinsing cleaned and decontaminated equipment with the organic-free water and submitting the rinse water to the laboratory for analysis. At least one rinse blank will be collected during each week (or 10 day event) of sampling operations. An attempt should be made to include as many of the same type of sampling for each rinse performed. This will help to ensure that a representative sampling is obtained. A rinsate should be collected from each type of sampling equipment being used. Any time a cleaning material different from those specified in the Cleaning and Decontamination Materials Section is used, an equipment rinsate sample must be submitted to the laboratory for analysis. 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.

Specified Field Equipment Cleaning and Decontamination Steps

Equipment used to collect samples that contain oil, grease, or other material difficult to remove may need to be rinsed several times with methanol or hexane before regular cleaning and decontamination steps are taken. In extreme cases, it may be necessary to steam clean the equipment. If the equipment cannot be adequately cleaned and decontaminated using these methods, it should be discarded.

Teflon® and Glass Field Sampling Equipment

1. Wash the equipment thoroughly with laboratory detergent and water using a brush to remove any particulate matter or surface film.
2. Rinse the equipment thoroughly with tap water.
3. If necessary (metals analyses), rinse the equipment with a 10 percent or stronger nitric acid solution. Small and awkward equipment, such as vacuum bottle inserts and well bailers, may be soaked in the nitric acid solution instead of being rinsed with it. Prepare fresh nitric acid solution for each cleaning.
4. Rinse the equipment thoroughly with tap water.
5. Rinse the equipment twice with pesticide-grade isopropyl alcohol and allow to air dry.
6. Rinse the equipment thoroughly with organic-free water.
7. Wrap the equipment completely with aluminum foil (dull side in) to prevent contamination during storage and/or transport to the field.

Stainless Steel or Metal Field Sampling Equipment

1. Wash the equipment thoroughly with laboratory detergent and water using a brush to remove any particulate matter or surface film.
2. Rinse the equipment thoroughly with tap water.
3. Rinse the equipment twice with pesticide-grade isopropyl alcohol and allow to air dry.
4. Rinse the equipment thoroughly with organic-free water and allow to air dry.
5. Wrap the equipment completely with aluminum foil (dull side in) to prevent contamination during storage or transport to the field. Larger pieces of equipment (e.g., auger flights with 5-foot split spoon samplers attached) may be wrapped in new Visqueen or the equivalent.

Specific Cleaning and Decontamination Steps for Sample Tubing

Silastic Rubber Pump Tubing (Automatic Samplers and Peristaltic Pumps)

New cleaned tubing is used for each automatic sampler setup. The silastic rubber pump tubing need not be replaced in peristaltic pumps where the sample does not contact the tubing or where the pump is being used for purging purposes (i.e., not being used to collect samples).

The silastic tubing is cleaned as follows:

1. Flush the tubing with tap water and phosphate-free laboratory detergent.
2. Rinse the tubing thoroughly with tap water.
3. Rinse the tubing with organic-free water.
4. Cap both ends of the tubing with aluminum foil (dull side in) until ready for use.

Teflon® Tubing (bladder pumps and small diameter electric pumps)

New Teflon® tubing, used for collection of samples for organic compound analyses, is cleaned as follows:

1. Cut the Teflon® tubing into convenient lengths before cleaning.
2. Rinse the outside of the tubing with pesticide-grade isopropyl alcohol.
3. Flush the interior of the tubing with pesticide-grade isopropyl alcohol.
4. Rinse the equipment thoroughly with organic-free water.
5. Wrap the equipment completely with aluminum foil (dull side in) to prevent contamination during storage or transport to the field.

Polyvinyl Chloride (PVC) Tubing (bladder pumps and small diameter electric pumps)

PVC tubing is used selectively and only where organic compounds are of no concern. Only new tubing is used. The tubing is flushed with sample immediately before use to remove residues from the manufacturing or extruding process. The tubing is stored in the original container and not removed until needed.

Stainless Steel Tubing

Stainless steel tubing is washed with laboratory detergent and water using a long, narrow bottle brush. Steps 2 through 6, as outlined in Section C.13.3.4, are then followed.

Glass Tubing

Only new glass tubing is used. The tubing is cleaned as follows:

1. Rinse the tubing thoroughly with pesticide-grade isopropyl alcohol.
2. Air dry the tubing.
3. Wrap the tubing completely with aluminum foil (dull side in) to prevent contamination during storage.

Specific Cleaning and Decontamination Steps for Miscellaneous Equipment Submersible Pumps and Hoses Used to Purge Groundwater Wells

1. Wash the equipment with laboratory detergent and tap water, running solutions through the pumps and pump hoses.
2. Rinse the equipment with tap water.
3. Rinse the equipment thoroughly with pesticide-grade isopropyl alcohol.
4. Rinse the equipment with organic-free water and allow to air dry.
5. Place the equipment in a polyethylene bag or wrap with polyethylene film to prevent contamination during storage or transit.

Well Sounders or Tapes Used to Measure Groundwater Levels

1. Rinse the equipment with pesticide-grade isopropyl alcohol.
2. Rinse the equipment with organic-free water.
3. Air dry the equipment.
4. Wrap the equipment completely with aluminum foil (dull side in) to prevent contamination during storage.

Drilling Rigs and Equipment

1. Before being mobilized and brought onsite, clean the engine and power head with a power washer or steam cleaner, or hand washed with a brush using detergent (does not have to be laboratory detergent but should not be a degreaser) to remove oil, grease, and hydraulic fluid from the exterior of the unit. Rinse these units thoroughly with tap water.
2. Steam clean and rinse all auger flights, auger bits, drilling rods, drill bits, hollow-stem augers, split-spoon samplers, Shelby tubes, or other parts of the drilling equipment that will contact the soil or groundwater prior to arriving onsite and between each boring.

Miscellaneous Sampling, Flow Measuring, and Field Instrumentation and Equipment

Miscellaneous flow measuring and sampling instrumentation is washed with laboratory detergent, rinsed with tap water, followed by a thorough deionized or organic-free water

rinse, and dried before being stored. This procedure does not apply to any equipment used for the collection of samples for trace organic compounds or metals analyses.

The exterior of sealed, watertight equipment, such as flow meters, should be washed with a mild detergent (e.g., liquid dishwashing detergent) and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth if necessary.

Other field instruments should be wiped with a clean, damp cloth; pH meter electrodes, conductivity electrodes, dissolved oxygen meter electrodes, etc., should be rinsed with deionized water before storage.

Ice chests and reusable shipping containers are washed with laboratory detergent (interior and exterior), rinsed with tap water and air dried before storage. In the event that an ice chest or shipping container becomes severely contaminated, it is cleaned as thoroughly as possible, rendered unusable, and disposed of properly.

Pressure Field Filtration Apparatus

The steps for cleaning Teflon® and glass equipment are used (Section C.13.3.4), except that the apparatus is assembled and pressure is applied after each rinse step to drive the rinse liquid through the porous glass filter holder in the bottom of the apparatus. After cleaning and decontamination, the apparatus is assembled and the pressure inlet and sample discharge lines are capped with aluminum foil (dull side in) to prevent contamination during storage.

Decontamination Procedures for Modified Low-Flow sampling

Refer to the modified low-flow sampling procedures discussed below. The following procedures will be followed to reduce contamination between sampling points during modified low-flow sampling.

1. All wells sampled via modified low-flow techniques will be equipped with dedicated, Teflon®-lined, HDPE tubing.
2. Prior to using new tubing, pump a deionized water rinse through the tubing and wash the tubing surface thoroughly with laboratory detergent and water, using a brush to remove any particulate matter or surface film. Rinse the tubing surface with isopropyl alcohol, followed by deionized water, and allow to air dry.
3. Decontaminate the submersible pump per decontamination procedures prior to each use.
4. Decontaminate the field parameter instrumentation prior to each use according to the procedures outlined above.
5. After each well has been sampled, pump a deionized water rinse through the tubing, and if necessary, wash the surface with laboratory detergent and water to remove any particulate matter or surface film. Rinse the tubing surface with isopropyl alcohol, followed by deionized water, and allow to air dry.
6. After the tubing has been allowed to air dry, place it in a polyethylene bag and label with the monitoring well ID.

7. Take periodic equipment rinses from the tubing in order to determine decontamination effectiveness and tubing integrity. At visible signs of tubing wear (staining, odor, excessive nicks and scrapes) or positive equipment rinse results, replace dedicated tubing.

Groundwater Sampling

Purpose and Applicability

This procedure conforms to the EPA Quality Assurance Requirements. It describes methods for purging and sampling a groundwater monitoring well to ensure that the sample collected is representative of the formation groundwater.

Definitions

Bailer: A hollow tube constructed of stainless steel or Teflon® that is used to collect groundwater samples. A dedicated bailer remains in the well casing.

Procedures

Purging

The following equipment is required for well purging:

1. Bailer or pump. The device used depends upon aquifer properties, individual well construction, well yield, and DQOs.
2. Water level measuring device.
3. Tape measuring device.
4. pH, specific conductance, turbidity, and temperature measuring device.

Well purging is performed as follows:

1. For the well to be purged/ sampled, obtain and record the following information on the groundwater purging/sampling data sheet or in the field log book: date, field conditions, well location, well ID, well diameter, groundwater elevation, total well depth, screened interval, water quality field measurements (pH, specific conductance, turbidity, and temperature), and the method for disposal of purged water.
2. Calibrate field instruments prior to use and according to manufacturer's instructions.
3. Prior to opening the well, place plastic sheeting on the ground surrounding the well head to prevent contamination by sample spillage.
4. Unlock and open the well and take an FID/PID reading immediately.
5. Measure the water level and the total depth of the well.
6. Calculate the volume in gallons of water in the well casing or sections of telescoping well casing as follows:

$$(\pi r^2 h) 7.48 = \text{gallons}$$

where: $\pi = 3.142$

r = Radius of the well pipe in feet

h = Linear feet of water in well

7.48 = Gallons per cubic foot of water

The volume of water in typical well casings may be calculated as follows:

$$\text{gallons/feet} \times \text{ (linear feet of water)} = \text{total gallons}$$

where:

2-inch well = 0.163 gallons/foot

3-inch well = 0.367 gallons/foot

4-inch well = 0.653 gallons/foot

5-inch well = 1.02 gallons/foot

6-inch well = 1.469 gallons/foot

7-inch well = 1.999 gallons/foot

8-inch well = 2.611 gallons/foot

10-inch well = 4.28 gallons/foot

12-inch well = 5.87

7. To purge the well, lower the decontaminated purging apparatus (pump or bailer) to the standing water column so that the water will be pulled through the casing and the entire static volume will be removed. Use a bailer when the well does not yield sufficient water for pumping; otherwise, a pump is preferred.
8. Measure the initial pH, specific conductance, turbidity, and temperature of water and record in the field logbook, along with the odor, color, clarity, silt concentrations, and general water condition. During purging, measure field parameters at least once during each well volume (more often is preferable). Record changes in the physical condition of the monitoring wells that could affect the well integrity.
9. For purging to be complete, remove at least 3-5 volumes of groundwater from the well, and allow the field parameters to stabilize. Measure the amount of purged fluid by filling a graduated bucket or using a stopwatch and noting the flow rate of the pump versus elapsed time. Stabilization for each field parameter is defined as follows: pH measurements ± 0.1 units, temperature measurements $\pm 1^\circ\text{C}$, specific conductance measurements ± 10 percent, and ± 10 percent for turbidity).
10. Purge wells with little or no recharge to near dryness, and allow the well to recover before sampling.
11. When using a pump prior to the completion of purging activities, bring the pump to the water surface to ensure complete removal of stagnant water.
12. Place purged water in a storage tank and disposed as IDW (as specified in IDW Plan).

Wells will be sampled immediately after purging, if possible, but generally no later than 6 hours after purging. Wells that recharge slowly will be purged dry and allowed to recharge before sampling. If excessive time (greater than 10 hours) is required for the slow recharging wells to recharge, it will be documented in the field log.

Sample Collection

Following are the general procedures for groundwater sampling along with methods for utilizing specific sampling devices and techniques.

General

1. With the exception of low-flow sampling, before samples are taken, purge the well.
2. Clean and decontaminate sampling equipment prior to the commencement of sampling activities. A new pair of disposable gloves will be worn at each location by sampling personnel.
3. Use pre-labeled, pre-cleaned sample bottles with preservative added to contain the groundwater samples. Volatile organic analysis (VOA) samples will be collected first, followed by other organic analyses.
4. As the sample is taken, tilt the sample container slightly to allow the water to run down the inside of the sample bottle with a minimum of splashing.
5. Leave adequate space in the bottle to allow for expansion, except for VOA vials, which are filled to overflowing and capped. Check VOA vials for air bubbles; if air bubbles are detected, carefully add more sample to the vial, taking care to minimize the loss of preservative.
6. Place samples in appropriate containers and pack with ice in coolers immediately after the sample is collected.
7. Measure pH, conductivity, temperature, and turbidity after sample bottles have been filled and record the measurements in logbook.

Bailer

A decontaminated Teflon® bailer can be used to remove groundwater samples from a well as follows:

1. Lower a decontaminated and properly secured bailer to the sampling interval from which the sample will be collected.
2. Allow the bailer to fill with a minimum of surface disturbance to prevent sample water aeration. When the bailer is raised, the bailer cord must not touch the ground.
3. Slowly pour the sample from the bailer, tilting the bottle slightly to allow the water to run down the inside of the sample bottle with a minimum of splashing.
4. If the bailer is dedicated, return it to the well and cap and lock the well. Clean and decontaminate nondedicated samplers after use.

Purging/Sampling Using Modified Low-Flow Techniques

Low-flow techniques are utilized to obtain a more representative sample from the aquifer formation. AGVIQ-CH2M HILL will use this procedure for the groundwater sampling at SWMU 54 and 55. In general, the advantages of low-flow purging include (EPA, 1996c):

- Samples which are representative of the mobile load of contaminants present (dissolved and colloid-associated)
- Minimal disturbance of the sampling point, thereby minimizing sampling artifacts (i.e. less turbidity)

- Less operator variability, greater operator control
- Reduced stress on the formation (minimal drawdown)
- Less mixing of stagnant casing water with formation water
- Reduces the need for filtration and thus the time needed for sampling
- Smaller purging volume which decreases IDW disposal costs
- Better sample consistency; reduced artificial sample variability

The pumps selected to perform low-flow sampling should be capable of producing purge rates sufficient to allow for the modified low-flow sampling technique. Pumps, which meet these requirements include, but are not limited to, bladder-type pumps (provided that reagent grade nitrogen is used for bladder inflation) and the Grundfos Redi-Flow2 pump.

Following are the procedures for modified low-flow groundwater sampling. These procedures include adaptations from EPA's paper entitled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (EPA, 1996c):

1. Slowly lower the decontaminated pump to the middle of the screened interval. This method will minimize the mixing of stagnant water in the casing above the screen with the screened interval zone water, and minimize re-suspension of solids which have collected at the bottom of the well.
2. Once the pump is positioned in the well, an airtight flow-through cell (equipped with a YSI or Horiba-type water quality meter) is plumbed to the water discharge line.
3. Lower a decontaminated water level gauge into the well to monitor the water table.
4. Once purging is initiated, water level measurements should be continuously monitored, and pumping rates adjusted as necessary (e.g., 0.1 - 0.3L/min) to maintain minimal drawdown. Modified low-flow techniques should cause less than 3 feet of drawdown during purging.
5. While purging, the groundwater field parameters (including water level) should be continuously monitored every 3-5 minutes until all parameters have stabilized for three consecutive readings.
6. Stabilization for each parameter is defined as follows: ± 0.1 for pH, ± 3 percent for conductivity, ± 10 mV for redox potential, ± 10 percent for turbidity, ± 10 percent for dissolved oxygen (DO), and ± 3 ft for drawdown.

Once field parameters have stabilized for three consecutive readings, samples may be taken. The same device used for purging should be used for sampling (remove flow-through cell).

Potassium Permanganate Injections

Purpose and Applicability

This procedure describes methods for potassium permanganate (KMnO₄) injections.

General

The KMnO₄ solution will be mixed onsite at a central mix station. The solution will be mixed to the maximum KMnO₄ concentration possible using site water. The KMnO₄ solution will be transported to the injection wells using transport trailers. **DO NOT LEAVE TRANSPORT TRAILERS UNATTENDED DURING FILLING OR INJECTIONS.** The ISCO injections consist of the following tasks:

- Mixing the KMnO₄ solution;
- Filling the transport tanks on the injection trailers;
- Injections in existing wells; and
- Monitoring during injections.

These steps are described in detail below.

Mixing the KMnO₄ Solution

The KMnO₄ solution will be mixed using the Carus™ Cycle Bin (Cycle Bin) and a 9,000-gallon Baker™ mix tank. The estimated KMnO₄ injection concentration will range from 30 to 40 g/L. To achieve this concentration, the KMnO₄ solutions will be mixed with approximately 8,000 gallons of water and 1 super sack (approximately 2,000 pounds) of KMnO₄. Mix time required to achieve KMnO₄ solubility will be based on field observations. The following steps will be followed to mix the KMnO₄ injection solution:

1. Plumb the backflow prevention assembly and water service meter to the water supply hydrant.
2. Ensure the water supply hydrant is plumbed correctly to the mix tank and the hydrant discharge valve is closed (Valve 1 on Figure 2-2).
3. Completely open the hydrant valve.
4. Note the reading on the hydrant flow meter and record in the Mix System Log.
5. Open the inlet valve (Valve 5 on Figure 2-2) on the mix tank.
6. Open the hydrant discharge valve (Valve 1 on Figure 2-2).
7. Fill the mix tank until the mixer blades are covered with water.
8. Start the mixers.
9. Use a forklift to place a KMnO₄ super sack on the Cycle Bin. **Note, only individuals with proper certification may operate the forklift.**
10. Open the inlet valve on the mix tank.

11. Open the sludge discharge valve (Valve 2 on Figure 2-2) on the Cycle Bin.
12. Open inlet valve (Valve 8 on Figure 2-2) to initiate flow of water into the Cycle Bin.
13. Turn on power to the Cycle Bin.
14. Simultaneously allow water from the hydrant and the Cycle Bin to fill the mix tank.
15. In sequence, close Valves 1, 5, 8, 6, and 2 when all the KMnO_4 in the Cycle Bin has been transferred to the mix tank and the mix tank contains approximately 8,000 gallons water.
16. Note the reading on the hydrant flow meter and record in the Mix System Log.
17. Continue stirring the mix tank until adequate mix time is reached.
18. Turn off mixers.

Filling the Transport Tanks

DO NOT LEAVE THE TRANSPORT TRAILER UNATTENDED DURING THE FILLING PROCESS. The process flow diagram for the transport trailer is shown on Figure 2-3. Note that all transfer hoses will be fitted with quick connection hardware. The transport tanks will be filled only with KMnO_4 solution from the mix tank--no other material may ever be put in the transport tanks. The following steps will be followed to fill the transport tanks:

1. Park the transport trailer on level ground, engage the parking brake, and place wheel chocks.
2. Connect the mix system discharge hose to the fill port for transport tanks.
3. Ensure all valves on the transfer trailer (Valves 1, 2, 3, 4, 5 on Figure 2-3) and on the transport tanks are closed.
4. Ensure transfer valve to trailer is closed (Valve 4 on Figure 2-2).
5. Open transfer pump valve and recirculation valve on mix system (Valves 7 and 3 on Figure 2-2).
6. Turn on the transfer/recirculation pump to establish recirculation in the mix tank.
7. Slowly open the transfer valve to trailer (Valve 4 on Figure 2-2), allowing flow into the transport tanks.
8. Watch the sight glass on the transport tanks to evaluate the fill rate.
 - a. If the fill rate is insufficient, slowly close the mix system recirculation valve (Valve 3 on Figure 2-2) until a reasonable fill rate is achieved.
 - b. If the fill rate is sufficient, allow the transport tank(s) to fill.
9. When the transport tanks are full, turn off the transfer pump.
10. In sequence, close the transfer pump valve (Valve 7 on Figure 2-2), recirculation valve (Valve 3 on Figure 2-2), and transfer valve to trailer (Valve 4 on Figure 2-2).

11. Disconnect the mix system discharge hose from the fill port. Ensure the discharge hose is inside the mix system secondary containment.
12. Ensure all valves on trailer are securely closed and there are no leaks.
13. Remove wheel chocks before attempting to move trailer.

KMnO₄ Injection

DO NOT LEAVE THE TRANSPORT TRAILER UNATTENDED DURING THE INJECTION PROCESS. A total of approximately 16,000 gallons of KMnO₄ solution will be injected at each well. The following steps will be followed to conduct the KMnO₄ injections:

1. Park the transport trailer on level ground, engage the parking brake, and set wheel chocks.
2. Remove well cap(s) and connect the trailer discharge hose(s) to the well head(s).
3. Either zero the flow meter(s) or note the totalizer volume using the Injection System Log (see Appendix C).
4. Open the injection system recirculation valve (Valve 3 on Figure 2-3).
5. Ensure that injection valves are closed (Valves 4 and 5 on Figure 2-3).
6. Open injection pump valves (Valves 1 and 2 on Figure 2-3).
7. Start the transfer pump, establishing flow back into the transport tank(s).
8. Slowly open first injection valve (Valve 4 on Figure 2-3) on one leg of the manifold and establish flow to one well.
9. Slowly open the first injection valve (Valve 4 on Figure 2-3) until the well head pressure is maximized at 30 psi. Make small, incremental adjustments and allow time for the pressure to equilibrate between adjustments. If necessary to increase well head pressure, partially close recirculation valve (Valve 3 on Figure 2-3).
10. While stabilizing injection flow, look around the injection area to check for surfacing of injection fluid. If KMnO₄ is surfacing, immediately turn off power to the injection pump, close all valves, and initiate spill response.
11. Slowly open the second injection valve (Valve 5 on Figure 2-3) on the other leg of the manifold and establish flow to the second well.
12. Slowly open the second injection valve (Valve 5 on Figure 2-3) until the well head pressure is maximized at 30 psi. Make small, incremental adjustments and allow time for the pressure to equilibrate between adjustments. If necessary to increase well head pressure, partially close recirculation valve (Valve 3 on Figure 2-3).
13. While stabilizing injection flow, look around the injection area to check for surfacing of injection fluid. If KMnO₄ is surfacing, immediately turn off power to the injection pump, close all valves, and initiate spill response.
14. Check pressure in first well. Maintain pressure by adjusting valve (Valve 4 on Figure 2-3).

15. When flow has stabilized at both wells, look around the injection area to check for surfacing of injection fluid. If KMnO_4 is surfacing, immediately turn off power to the injection pump, close all valves, and initiate spill response.
16. Frequently monitor the KMnO_4 solution level in the transport tank(s) – DO NOT allow the tank(s) to drain completely. Operation of the injection pump without liquid will burn up the pump.
17. Continuously monitor the area for signs that injection solution is surfacing. If KMnO_4 is surfacing, immediately turn off power to the injection pump, close all valves, and initiate spill response.
18. Record injection flow rates, totalizer volume, and pressures approximately every 30 minutes on the Injection Log.
19. When the transport tank(s) need to be refilled, turn off the injection pump, close all valves and check for leaks.
20. Disconnect the discharge lines from the trailer manifold and place the ends in 5-gallon buckets at each well.
21. Remove wheel chocks before attempting to move trailer.
22. Upon completing injection at a well, disconnect discharge line from well head and secure well cap.

Spill Response

General

Four chemicals will be used during the ISCO injections: KMnO_4 , sodium thiosulfate ($\text{Na}_2\text{O}_3\text{S}_2$), hydrogen peroxide (H_2O_2), and acetic acid (vinegar). Potassium permanganate will be present in two forms: solid (KMnO_4 powder) and aqueous solution of 30 to 40 g/L KMnO_4 solution. The remaining chemicals, $\text{Na}_2\text{O}_3\text{S}_2$, H_2O_2 , and vinegar are used to neutralize KMnO_4 drips and spills. The $\text{Na}_2\text{O}_3\text{S}_2$ will also be present in two forms: solid ($\text{Na}_2\text{O}_3\text{S}_2$ powder) and aqueous solution of approximately 6 percent $\text{Na}_2\text{O}_3\text{S}_2$ solution. This section describes spill response procedures for each of these four chemicals. The material safety data sheets (MSDS) for each chemical are provided in Appendix D.

Neutralization of KMnO_4 solutions is required under three circumstances: 1) drips and small spills of injection solution, 2) large spills of injection solution, and 3) handling of purge water during post-injection monitoring. Drips and small spills (less than 1 gallon of KMnO_4) should be neutralized by spraying the area with a 1 percent H_2O_2 solution. The 1 percent H_2O_2 solution is made from equal volumes of 3 percent H_2O_2 , vinegar, and water. Larger spills (greater than 1 gallon KMnO_4) should be neutralized by spraying the area with a 6 percent sodium thiosulfate ($\text{Na}_2\text{O}_3\text{S}_2$) solution, made of 55 gallons water and 27.5 pounds sodium thiosulfate. In both cases, the neutralizing agent (either H_2O_2 or $\text{Na}_2\text{O}_3\text{S}_2$) should be sprayed on the spill area and allowed to react for 2 minutes before additional application. The person conducting the neutralization should ensure that the KMnO_4 and the neutralizing agent are fully mixed AND that sufficient reaction time has elapsed

(approximately 2 minutes or until there is no longer evidence of change in color) before adding more material.

If excessive gas or steam generation is observed during neutralization, STOP adding neutralizing chemical and add water. Allow the heat to dissipate before addressing the spill further.

KMnO₄ Solution Spill Response

- Clear personnel from the spill area.
- Dress in protective face shield and chemical-resistant clothing.
- Contain spill with soil berms or other chemically compatible materials.
- Neutralize the KMnO₄ solution with a 6 percent Na₂O₃S₂ solution, according to the general method described above, until spill is fully contained and neutralized.

If personnel are exposed to KMnO₄ solution, thoroughly rinse all affected areas with water immediately. Consult the KMnO₄ MSDS (Appendix D) for first aid procedures.

KMnO₄ Solid Spill Response

- Clear personnel from the spill area.
- Clean up spills immediately by sweeping or shoveling up the material. Transfer spilled material to a clean metal drum. Do not return spilled material to the original container. Flush the contaminated surface with water.
- If necessary, neutralize the contaminated surface with a 6 percent Na₂O₃S₂ solution, according to the general method described above, until spill is fully contained and neutralized.

Na₂O₃S₂ Solution Spill Response

- Clear personnel from the spill area.
- Remove metal tools from the area.
- Contain spill with soil berms or other chemically compatible materials.
- Dilute the spilled solution with water (approximately 10 volumes of water per volume of Na₂O₃S₂ solution).
- The diluted solution may be rinsed into the water treatment system or storm drain. Otherwise, the diluted solution may be containerized for disposal offsite.

If personnel are exposed to Na₂O₃S₂, thoroughly rinse all affected areas with water immediately and consult the Na₂O₃S₂ MSDS (see Appendix D) for first aid procedures.

Na₂O₃S₂ Solid Spill Response

- Clear personnel from the spill area.

- Clean up spills immediately by sweeping or shoveling up the material. Transfer spilled material to a clean plastic drum. Do not return spilled material to the original container. Flush the contaminated surface with water.

H₂O₂ Spill Response

- Clear personnel from the spill area.
- Dilute the H₂O₂ spill with water.
- The diluted solution may be rinsed into the water treatment system or storm drain. Otherwise, the diluted solution may be containerized for disposal off-site.

If personnel are exposed to H₂O₂, thoroughly rinse all affected areas with water immediately and consult the H₂O₂ MSDS (see Appendix D) for first aid procedures.

Vinegar Spill Response

- Clear personnel from the spill area.
- Dilute the vinegar spill with water.
- The diluted solution may be rinsed into the water treatment system or storm drain. Otherwise, the diluted solution may be containerized for disposal off-site.

If personnel are exposed to vinegar, thoroughly rinse all affected areas with water immediately and consult the vinegar MSDS (see Appendix D) for first aid procedures.

Personal Protective Equipment

The PPE required for site activities is a minimum of level D. This includes long pants, steel-toed safety shoes, and safety glasses. When there is risk workers may be exposed to the KMnO₄ injection solution, for example during spill response, workers should don face shields, chemical aprons, and chemical gloves. Rubber boots are also recommended. Additional PPE requirements are explained in the H&S (Appendix D of the PAWP) and the AHAs presented in Appendix A of this document.

Logs and Record Keeping

In addition to the site log book (maintained by the Field Team Leader), logs and forms will be completed in association with the ISCO injection activities:

- Mix System Log
- Injection System Log
- Daily Report
- Pre-Task Safety Plan
- Safe Behavior Observation Form
- Material Receipt Form

Analytical SOPs from Worksheet #23

GULF COAST ANALYTICAL LABORATORIES, INC.
WET LAB
STANDARD OPERATING PROCEDURE

PROCEDURE: WL-060
PAGE: 1 OF 5
EFFECTIVE DATE: 09/29/2008
APPROVED BY: *MWP*
QA/QC APPROVED: *JDT*

SUBJECT SCOPE AND APPLICATION

An automated Pensky-Marten closed cup tester is utilized to determine the flashpoint of both liquid and solid samples. Solids samples, however, do not fall within the scope of this method because of the inability to stir and uniformly heat the sample.

SUMMARY The sample is heated at a slow, constant rate (9-11°F/min) with continual stirring (105rpm). As the sample is heated, a small electric heated coil is directed into the cup every 2°F with simultaneous interruption of stirring until a flashpoint is detected by the thermal detector. By definition, the flashpoint is the lowest temperature at which the vapors above the sample will ignite when subjected to a source of ignition.

MATRIX Water and Solid

REFERENCE SW-846, 1010A
ASTM D93

PRESERVATIVE Samples should be stored at 4°C with a minimal amount of headspace in the container. When possible, a separate container should be allocated for flashpoint analysis only.

HOLDING TIME Not specified by method

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and the supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures, solvents must be handled in ventilated hoods.

APPARATUS Herzog MP-330-Automated Pensky Marten Closed Cup Flashpoint Tester
Fire extinguisher
PT-100 thermometer
Thermal detector
Electric igniter
Brass cup with lid
Stirring cable

Disposal container labeled flashpoint waste
Aluminum pans
Matches or lighter

REAGENTS

o-Xylene (QC); known flashpoint of 90°F

PRE-ANALYSIS
PROCEDURE

1. Use a thermometer to determine the ambient temperature of the room. Record the temperature in the flashpoint logbook.
2. All samples should be tested for ambient flashpoint before being loaded onto the sample apparatus.
3. Place a small amount (approximately 2ml or 2g) of sample in an aluminum pan.
4. Pass a lighted match or burning lighter 1 to 1 ½ inches above the sample. Do not touch flame to sample.
5. If the sample vapors ignite, the result should be recorded as ambient in the flashpoint logbook and reported as <ambient temperature in the LIMS.
6. If the sample does not ignite, proceed to the next section in this Standard Operating Procedure.

PROCEDURE

A. Liquid Samples

1. Shake samples thoroughly and fill brass cup to the line. This should take approximately 75 mls of sample.
2. Place brass cup with sample in the heating block so that the three circular keys fit into the corresponding slots.
3. Turn the locking arm to the left to lock the brass cup into position.
4. Place the cup lid on the brass cup so that the rectangular key fits in the cover slot and is snug.
5. Carefully insert the PT-100 thermometer and thermal detector into the correct slots on the cup lid.
6. Check the igniter filament for damage. It should have fairly uniform distance between the coils. Replace if damaged.
7. Place the drive rod arm into the appropriate slot on the top of the cup.
8. Attach the stirring cable to both the cup lid and the stirrer coupling.
9. From the instrument display, press the "P" button until the light for Program 4 is lit.

NOTE: This is a screening program and should only be used when the flashpoint is unknown. If the flashpoint of the sample is already known, proceed directly to step 22 in this Standard Operating Procedure.

10. From the instrument display, press, "start" so that the word SAMPLE NUMBER appears.
11. Type in the appropriate sample number or sample ID and once again press, "start" on the instrument display.
12. At this point the instrument display will ask for an expected flashpoint. For unknown samples, use the key pad on the instrument display to type in the number 140°F. This allows the instrument to screen the sample over the range from ambient to 212°F.
13. Once again, press, "start". The instrument should display the barometric pressure. Press, "start". The phrase "START TEST?" should appear. Press, "start" one final time to start the analysis.
14. At this point the instrument should perform a series of checks. The analyst should hang around long enough to verify that the igniter coil is working and the drive rod arm is detecting the proper zero position.
15. Once the flashpoint has been detected or the instrument reaches 212°F without detecting a flashpoint, the analysis will stop and the analyst will be alerted with an alarm.
16. To turn off the alarm, the analyst should hit the "Stop" button.
17. Immediately following the analysis, the cooling system will turn on. Once the instrument is cooled to an acceptable level, once again the alarm will be sounded and the analyst should hit the "Stop" button to turn it off.
18. The test result shown in the display should be recorded in the flashpoint logbook.
19. If the instrument reaches 212°F without detecting a flashpoint, the result should be recorded as >212°F. If this is the case, the analyst should move on to the next sample.
20. If a flashpoint is detected below 212°F, it must be verified.
21. At this point, the apparatus should be carefully taken apart and all parts cleaned with a suitable solvent (deionized water, acetone, methanol, etc.).

22. To verify a flashpoint or run a known flashpoint, a fresh aliquot of sample should be used.
23. Set up a fresh aliquot of sample using steps 1 through 8 in this Standard Operating Procedure.
24. From the instrument display, press "P" button until the light for ISO/ASTM-A is lit. Any flashpoints that are to be confirmed should be run in this program.
25. Follow steps 10 through 18, except in step 12 enter the known flashpoint instead of 140°F.
26. The analysis is complete if the two runs yield results that are within $\pm 9^\circ\text{F}$ of each other. If this criterion fails, a third analysis should be performed in the ISO/ASTM-A program mode.

B. Solid Samples

Same as liquids above except for the following:

1. Samples should be homogenized with a spatula.
2. Loosely fill brass cup to line with solid sample.
3. Do not attach the stirring cable.
4. Results should fall within $\pm 25^\circ\text{F}$ to be accepted.

CALCULATIONS No calculations are required.

DETECTION LIMIT The maximum value reported for flashpoint at Gulf Coast Analytical Laboratories, Inc. is 212°F. Because of the purpose of analyzing the flashpoints at GCAL, it is impractical to continue past this temperature. Any sample that does not flash before this temperature should be reported as $>212^\circ\text{F}$ in both the logbook and the LIMS.

QUALITY CONTROL

1. One QC sample should be analyzed per batch of 20 samples. o-Xylene is used as this standard and has a known flashpoint of 90°F. The control limits for this QC sample is $90 \pm 2^\circ\text{F}$. If the above control limits are not met, no samples can be analyzed. Retest the o-Xylene a second time. If the second analysis of the o-Xylene meets the control limits, then sample analysis can proceed. If the second analysis fails the control limits, a fresh container of o-Xylene must be obtained before any further analysis may proceed.
2. Any sample that has a flashpoint below 212°F should be duplicated. Liquid sample duplicate flashes should agree within $\pm 9^\circ\text{F}$, while solid samples should agree within $\pm 25^\circ\text{F}$.

3. The second run, the one that uses the ISO/ASTM-A program, is the run that should be reported.

METHOD
PERFORMANCE

Repeatability - The difference between successive results, obtained by the sample operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values in 1 case in 20.

$$r = AX,$$

$$A = 0.029,$$

X = mean result in °C, and

r = repeatability

Reproducibility - The difference between two single and independent results, obtained by different operators working in different laboratories on identical material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

$$R = BX,$$

$$B = 0.071,$$

X = mean result in °C, and

R = reproducibility

Bias - Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

Relative Bias - Statistical evaluation of the data did not detect any significant difference between the reproducibility variances of manual and automated Pensky-Martens flashpoint results for the samples studied. Evaluation of the data did not detect any significant difference between averages of manual and automated Pensky-Martens flash point for the samples studied with the exception of cycle oil and fuel oil that showed some bias. In any case of dispute, the manual procedure shall be considered the referee test.

NOTE: The precision statements were derived on clear liquids only. Refer to the research report⁹ for information regarding relative bias and types of samples. Additional studies are in progress concerning relative bias.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC
EXTRACTIONS
STANDARD OPERATING PROCEDURE

PROCEDURE: EXT-017
PAGE: 1 OF 6
EFFECTIVE DATE: 02/20/09
APPROVED BY: *MAB*
QA/QC APPROVED: *JDT*

SUBJECT SCOPE AND APPLICATION

This procedure is designed to describe the steps for the preparation of aqueous samples and TCLP extracts for the analysis of Extractable Herbicides.

The samples are extracted using a solid phase cartridge speed disk. After conditioning the disk samples are aspirated through the cartridge at low vacuum. Samples are eluted from the disk using ethyl acetate. The eluted sample is esterified with diazomethane. After excess reagent is removed, the esters are determined by gas chromatography.

MATRIX Water

REFERENCE SW846 8151A
SW846 3535A
JT Baker Application Note

PRESERVATIVE Cool to 4°C

HOLDING TIME From collection to extraction - 7 days
From extraction to analysis - 40 days

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

REAGENTS All organic solvent shall be of HPLC grade or equivalent. Reagents shall be of reagent grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.

Concentrated Hydrochloric acid
Methanol
DI Water
Acidified water - Add HCl to DI water until the pH is approximately 2
Ethyl acetate
Diazald
Diethyl ether

Potassium Hydroxide
Ethyl Alcohol

STANDARDS

All standards used are pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

Surrogate - Stock Standard: 200 ug/mL, 2,4-Dichlorophenylacetic Acid (DCAA) in Acetonitrile and stored at room temperature. Prepare a working standard (20 ug/mL) by adding 5 mL of the stock standard to a 50 mL volumetric flask and diluting to volume with Acetonitrile. The working standard may be held for six months or until expiration of the stock standard. Store at room temperature.

Spiking Standard - Commercially prepared Underivatized Chlorinated Herbicides Stock Standard - 100 ug/mL in Acetone and stored at 4°C. Prepare a working standard at a concentration of 10 ug/mL in methanol by adding 1mL of stock standard to a 10mL volumetric flask and diluting to volume in methanol. The working standard may be held for six months or until expiration of the stock standard. Store at 4°C.

APPARATUS

JT Baker Speedisk H₂O-Phobic - Test each lot of disks by performing an LCS and MB before extraction of samples. Manifold with vacuum that can maintain 25in of mercury

Liter amber bottles

VOA vials 44mL

1000mL graduated cylinder

10mL graduated cylinder (Class A)

Vials: Amber glass, 1-2 mL capacity with Teflon-lined screw cap

Water bath/Nitrogen stream: With temperature and gas flow control. The bath is used in a hood.

1 mL pipettor

Pasteur pipets

Wide-range pH paper

PROCEDURE

Sample Extraction for Water

1. Using a calibrated graduated cylinder, measure 1000mL of DI water for the Method Blank (MB), LCS, and LCSD. Pour into labeled 1L amber glass bottles. The MB and LCS/LCSD are labeled with a GCAL ID (these numbers are assigned by the LIMS). If sample is available perform an MS and MSD with each batch.
2. Acidify all QC and samples to a pH of ≤ 2 using HCl acid and wide range pH paper.
3. Spike each sample with 1.0 mL surrogate working standard. Spike the LCS/LCSD and MS/MSD with 1.0mL of the spike working standard.
4. Cover and shake each sample.

5. Condition speed disk by performing the following steps in order:
 - a. Pour 6mL of methanol into disk and pull through. Repeat with an additional 6mL.
 - b. Pour 6mL of DI water into disk and pull through.
 - c. Pour 6mL of acidified water into disk and pull through.
 - d. After each addition, pull the liquid through the disk but do not allow the disk to run dry between and after each step.
6. With the vacuum off add approximately 6mL of sample to disk. Attach adaptor and reservoir and add the sample.
7. Aspirate the sample through the disk slowly. The sample should drain through in approximately one hour.
8. Dry the disk under vacuum for 30 minutes. Check the apparatus for any water condensation. If any is found dry with a Kim wipe before performing sample elution.
9. Set-up the apparatus for sample elution into the VOA vial. Rinse the sample bottle with 6mL of ethyl acetate and pour into disk. Allow to soak for 10 minutes.
10. Elute the ethyl acetate into the VOA vial slowly. Repeat with a second aliquot of ethyl acetate.
11. Pour the sample into a glass tube.
12. Evaporate the sample slowly under a stream of nitrogen to 0.5mL.
13. Dilute the extract with 1 mL of isooctane and 0.5 mL of methanol. Dilute to a final volume of 4 mL with diethyl ether.
14. Add 2mL of Diazomethane to each sample to derivatize the Methyl esters. Allow the sample to stand for 10 minutes with occasional swirling. Verify the extract turns yellow upon adding the Diazomethane.
15. Add 10mg of Silicic acid to each extract to destroy the excess Diazomethane and allow to stand for 10minutes with occasional stirring.
16. Pour the sample into a class A graduated cylinder or 10mL volumetric. Add 5ml of Ethyl ether and dilute to final volume with hexane. There should not be visible layers in the diluted sample. If any are noted, document on prep sheet and notify supervisor.
17. Bottle 1 ml of sample in a crimp top amber vial and transfer to the Semi-volatile GC section with a copy of the prep log.
18. Samples are analyzed using SOP GC-011.

PROCEDURE

Diazomethane Generation

Caution: Diazomethane is a known carcinogen and can explode violently under certain conditions.

- The entire apparatus should be contained in a hood.
- DO NOT heat above 90°C - An explosion may result.
- DO NOT use any type of ground glass joints, glass stirring rods, or grinding surfaces - An explosion may result.
- KEEP AWAY from alkaline metals (Na, Mg, etc.) An explosion may result.
- There should be NO solid materials (boiling chips, sediment, etc) present in the samples or generator.

Diazomethane decomposes rapidly in the presence of solids and poses an explosion hazard.

PROCEDURE

Caution: ONLY experienced personnel shall operate the Diazomethane generator. Under NO circumstances shall an inexperienced person be allowed to operate it.

1. Assemble the generator. Make sure that the gas trap and collection flasks are surrounded with an ice bath.
2. Dissolve 20 grams of Diazald in 200 ml of Diethyl ether and shake well.
3. Dissolve 20 g of KOH into 32 ml of water and add 40 ml of Ethanol. Place in an amber bottle and cap.
4. Place 90 ml of the Ether/Diazald mixture in the separatory funnel on the generator with the stopcock closed. Place 40 ml of the water/Ethanol/KOH mixture in the reaction flask and submerge in a water bath that is approximately 65°C. Allow the system to come to equilibrium for about 20 minutes.
5. Start the generator by SLOWLY opening the stopcock on the separatory funnel until the ether solution begins dripping into the reaction flask. The optimum flow rate is about 60 drops per minute.
6. As the reaction progresses, a yellow liquid will begin to collect in the collection flask. Once all the Ether/Diazald mixture has been exhausted from the funnel, fill it with ten ml of ether and allow it to distill as well.
7. Turn off the hot water bath, and disassemble the generator. The Diazomethane/Ether solution is now ready to use.
8. Document Diazomethane generation in logbook and label the solution. Store sealed at 4°C.

METHOD PERFORMANCE

- 1) In single laboratory studies using organic-free reagent water and clay/still bottom samples, the mean recoveries presented in Table 4 and 5 of Method 8151A were obtained for diazomethane derivatization. The standard deviations

of the percent recoveries of these measurements are also in Tables 4 and 5.

- 2) Table 6 of Method 8151A presents relative recoveries of the target analytes obtained using the PFBB_r derivatization procedure with spiked water samples.

POLLUTION PREVENTION See QAPP Section 13.2

WASTE MANAGEMENT See SOP GEN-009

GULF COAST ANALYTICAL LABS
GC - CHLORINATED HERBICIDES
STANDARD OPERATING PROCEDURE

PROCEDURES: GC-011
PAGE: 1 OF 12
EFFECTIVE DATE: 01/30/08
APPROVED BY: *dlb 3/10/08*
QA/QC APPROVED: *[Signature]*

SUBJECT

SCOPE AND APPLICATION

This method provides gas chromatographic procedures for the detection of ppb levels of certain chlorinated herbicides. Prior to the use of this method, appropriate extraction techniques must be used. An aliquot of the sample extract is injected into a GC equipped with an ECD detector.

This SOP provides for the analysis of single component analytes. The QC requirements vary depending upon which analysis is being performed. Analysts should carefully read the SOP in order that requirements are not confused.

MATRIX

Water and Solid

REFERENCE

SW846 Method 8151A, 8000C

PRESERVATIVE

Cool 4°C

HOLDING TIME

Water - From collection to extraction - 7 days
From extraction to analysis - 40 days
Solid - From collection to extraction - 14 days
From extraction to analysis - 40 days

DEFINITIONS

See SOP GEN-016

SAFETY

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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

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.

INTERFERENCES

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.

INSTRUMENTATION

Gas Chromatograph should be suitable for splitless or on-column injection. The system should be equipped with an electron capture detector. The instruments used are Agilent Technology 6890N GC/ECD. The data system must be capable of time stamping, all data produced, with the correct date and time. The data system used for acquisition is Target.

COLUMNS

Recommended Columns are

XLB or similar phase	30m X 0.32mm ID
35MS or similar phase	30m X 0.32mm ID

REAGENTS

Hexane-pesticide grade. Store away from sources of phthalates.

STANDARDS

1. Standards: All standards are purchased certified standards in sealed ampules. The stock standards are prepared in MtBE or Hexane. The standards are stored at 4°C until the manufacturer's expiration date. The stock standards are the methyl esters.
2. ICV Standard: Stock standard solution containing all analytes of interest prepared from a source independent of the calibration standards.

Preparing Calibration Standards:

1. The calibration standards are prepared by diluting the stock solution into 5 or 6 working levels. The standards are prepared prior to use. The following chart indicates the compounds and concentrations.

TABLE 1
Concentrations ($\mu\text{g/L}$)

COMPOUND	Level 1 $\mu\text{g/L}$	Level 2 $\mu\text{g/L}$	Level 3 $\mu\text{g/L}$	Level 4 $\mu\text{g/L}$	Level 5 $\mu\text{g/L}$	Level 6 $\mu\text{g/L}$
2,4-D	11.8	23.6	47.2	94.3	188.7	70.8
2,4,5-T	11.8	23.6	47.2	94.3	188.7	70.8
2,4,5-TP Silvex)	11.8	23.6	47.2	94.3	188.7	70.8
2,4-DB	11.8	23.6	47.2	94.3	188.7	70.8
3,5-Dichlorobenzoic Acid	11.8	23.6	47.2	94.3	188.7	70.8
Acifluorfen	11.8	23.6	47.2	94.3	188.7	70.8
Bentazon	11.8	23.6	47.2	94.3	188.7	70.8
Chloramben	11.8	23.6	47.2	94.3	188.7	70.8
Dalapon	11.8	23.6	47.2	94.3	188.7	70.8
DCPA	11.8	23.6	47.2	94.3	188.7	70.8
Dicamba	11.8	23.6	47.2	94.3	188.7	70.8
Dichloroprop	11.8	23.6	47.2	94.3	188.7	70.8
Dinoseb	11.8	23.6	47.2	94.3	188.7	70.8
MCPA	1179	2360	4717	9434	18868	7080
MCPP	1179	2360	4717	9434	18868	7080
4-Nitrophenol*	11.8	23.6	47.2	94.3	188.7	70.8
Pentachlorophenol**	11.8	23.6	47.2	94.3	188.7	70.8
Picloram	11.8	23.6	47.2	94.3	188.7	70.8
DCAA	11.8	23.6	47.2	94.3	188.7	70.8

*methyl derivative of 4-Nitroanisole (name on certificate of analysis)

**methyl derivative of Pentachloroanisole (name on certificate of analysis)

2. All calibration standards reflect concentrations adjusted for molecular weight of the methyl ester versus the acid herbicide.
3. All standards should be made with Pesticide Grade Hexane.

RETENTION TIME STUDY 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.

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.

INITIAL CALIBRATION CURVE

The initial calibration consists of a series of standards analyzed at the concentration noted in Table 1. An initial calibration curve for each target analyte must be analyzed and evaluated before any peak for that analyte can be quantitated.

The calibration range is defined as the on-column concentration range adjusted for sample prep. If more than five 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:

- a) At least five continuous points must be used (six if a quadratic curve fit is employed).
- b) Calibration acceptance criteria must be met as described below.
- c) The lowest calibration point must support the lowest reporting limit needed in the associated samples.
- d) The QC spike amount must be within the calibration range.

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.

The calibration curve is now ready for analysis.

The acceptance criteria for initial calibration must be satisfied before analysis of samples begin. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.

1. Additionally one of the following options must be met. Always attempt to meet calibration criteria using the average response factor. If the average response factor does not pass, options B and C 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). The calibration options and requirements are as follows:

- A. 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.
- B. 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 must be at least 0.995 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.
- C. 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.

INITIAL CALIBRATION VERIFICATION

Immediately following the initial calibration procedure or before sample analysis, the analyst shall perform initial calibration verification (ICV). This will consist of a solution containing all target analytes prepared from a standard that is independent from the initial calibration.

The ICV must be analyzed following the initial calibration. The ICV recovery must be 85-115%. Specific project criteria may apply that must be achieved for the associated samples. See SOP GEN-019.

If any target analyte recovery is outside the control limits, 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.

CONTINUING CALIBRATION
VERIFICATION STANDARDS

1. The calibration verification standards are the mid-level standard of the calibration. The calibration verification standards are analyzed to verify that the calibration curve and retention time windows are still valid.
2. The calibration verification standard is analyzed at the beginning of each 12-hour shift. The shift begins with the injection of the calibration verification standard and ends after the completion of analysis of the last sample or standard injected within 12 hours of the beginning of shift. A calibration verification standard must also be injected every 10 samples.
3. The criteria for the calibration verification standard is that all analytes must be ± 20 Percent Difference (%D) from the initial calibration. For each CCV, each compound must fall within the retention time window.
4. If the average of the responses for all analytes are within 20%, then the calibration has been verified; however, the % difference cannot exceed 30% for an individual analyte. Include the failure in the report narrative. This option is not allowed for some projects including, but not limited to, DOD, AFCEE, Marathon LLC, and projects from or reported to South Carolina. Check with the project manager if this option is used. See SOP GEN-019.
5. If the calibration verification standard fails to meet these criteria, repeat the injection of the standard. If the standard continues to fail, take appropriate corrective action (inspection of GC, re-prep, standard, etc.) If these criteria cannot be met, a new calibration curve shall be prepared.
6. If the calibration verification standard analyzed after a group of samples has a response for an analyte $>20\%$ 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. If an analyte was not detected in the sample and the standard response is less than 20% below the initial calibration response, then re-injection is necessary.

SAMPLE ANALYSIS

1. Up to 10 samples (not including MB, LCS/LCSD) can be analyzed after the calibration verification standard.
2. All samples are evaluated on a primary column then a secondary column if an analyte above detection limit was detected. The secondary column confirms the presence of that analyte.
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 unless apparent coelution is present. (The lower value maybe reported if required by the project.) If the difference between the two results, calculated as an RPD, is greater than 40%, narrate the sample results. The results are flagged in the report.

SAMPLE
RE-EXTRACTION

Samples are to be re-extracted due to failed QC or due to the sample results. When a MB, LCS, or LCSD fails to meet criteria, the entire batch is sent to extractions for re-extraction. If the MS, MSD, or surrogates fail criteria, only the affected samples are sent to extractions. Particular samples may be re-extracted if the sample results do not match historical values, if a sample and a duplicate do not match, or if physical differences are noted in samples. If samples are re-extracted outside of method specified holding times, both analyses are reported. To request sample/batch re-extraction, do the following:

1. If the extract has been analyzed, process the file and load to LIMS.
2. Enter the code "RP" into the analysis code; this will schedule new sample prep.
3. Complete the Re-extraction Request and Tracking Form (attached) and submit a copy to extraction supervisor.
4. When new extracts are brought to the lab, complete the original Re-extraction Request and Tracking Form and report the data appropriately.
5. If additional sample is not available to re-prepare, check with project manager to determine appropriate action to report the available data.
6. If two sets of data will be reported, see login to obtain a re-extracted sample number.

CALCULATIONS

1. $MS \% REC = \frac{MS\ Concentration - Sample\ Concentration}{MS\ Concentration} \times 100$

spike added

$$2. \text{MSD \% REC} = \frac{\text{MSD Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$3. \% \text{ RPD} = \frac{\text{MS} - \text{MSD}}{(\text{MS} + \text{MSD}) / 2} \times 100$$

LCS/LCSD results are substituted to calculate %RPD between LCS and LCSD. Results are calculated using the concentration (not percent recovery).

$$4. \text{Response Factor} = A_s / C_s$$

A_s = Peak Area of analyte or surrogate

C_s = Concentration of the analyte or surrogate

$$5. \text{Surrogate/LCS Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

$$6. \text{Concentration using RF:} \\ \text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(D)(V_t)}{(\text{RF})(V_s)}$$

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(D)(V_t)}{(\text{RF})(W_s)}$$

A_s = Area of peak for the analyte in sample

D = Dilution factor

RF = Mean Response factor from initial calibration

W_s = Weight of sample extracted in g

V_t = Total volume of concentrated extract in μL

V_s = Volume of aqueous sample extracted in mL

$$7. \text{Concentration using linear curve fit:}$$

$$\text{Concentration } (\mu\text{g/L}) = [m(A_s) + b]D$$

$$\text{Concentration } (\text{mg/kg}) = [m(A_s) + b]D(5/W_s)$$

m = Inverse of slope

A_s = Area of peak for the analyte in sample

b = Intercept of the y-axis

D = Dilution factor

W_s = Weight of sample extracted

$$8. \text{Concentration using a quadratic curve fit:}$$

$$\text{Concentration } (\text{mg/L}) = [b + a(A_s) + a_2(A_s)^2]D(5/V_s)$$

$$\text{Concentration } (\text{mg/kg}) = [b + a(A_s) + a_2(A_s)^2]D(5/W_s)$$

$$9. \% \text{ Difference} = [(RF_1 - RF_c) / RF_1] \times 100$$

RF_1 = Average response factor from initial calibration

RF_c = Response factor from current verification check standard

$$10. \% \text{ Drift} = \frac{(\text{Calculated Conc.} - \text{Theoretical Conc.})}{\text{Theoretical Conc.}} * 100$$

$$11. \% \text{ RSD} = (\text{SD}/\text{X}) * 100$$

RSD = Relative Standard Deviation

X = mean of initial RF's for a compound

SD = Standard Deviation of average RF's for a compound

REPORTING LIMITS

The following chart indicates reporting limits for Chlorinated Herbicides Method 8151:

COMPOUNDS	WATER (ug/L)	SOLID (ug/kg)
2,4-D	1.5	20
2,4,5-T	0.5	17
2,4,5-TP (Silvex)	0.5	17
2,4-DB	5	167
3,5-Dichlorobenzoic Acid	1	100
Acifluorfen	1	100
Bentazon	1	100
Chloramben	1	100
Dalapon	10	330
DCPA	1	100
Dicamba	0.5	17
Dichloroprop	5	167
Dinoseb	3	83
MCPA	500	1670
MCPP	500	1670
4-Nitrophenol	1	33
Pentachlorophenol	1	33
Picloram	1	33

NOTE: Reporting limits may vary according to specific project requirements.

QUALITY CONTROL

METHOD BLANK

1. The method blank (extraction blank) is analyzed to demonstrate the extraction procedure did not introduce contamination.
2. No target analytes should be detected in the method blank above the reporting limit. If any target analytes are detected, data may not be reported and samples must be re-extracted and re-analyzed unless the following apply.
 - A. If a target analyte is detected above the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
 - B. If a target analyte is detected in the method

blank but there are no hits in the samples, the data may be reported with a narrative.

- C. Additional project criteria may apply. See SOP GEN-019.

SURROGATES

1. The surrogate is used to verify that each sample was properly extracted. A surrogate is a non-target compound that is chemically similar to the analytes. The surrogate use is 2,4-Dichlorophenylacetic Acid (DCAA).
2. If a surrogate recovery fails below the lower control limit, re-extract and re-analyze the sample. If the surrogate is outside QC limits in the re-extract sample, then indicate in the case narrative and state that the recovery was outside the control limits due to sample matrix. If re-extraction cannot be performed due to insufficient sample, report the data with a narrative.
3. If the surrogate recovery is above the control limits and the sample results are less than the reporting limit, the data may be reported with a narrative.

<u>Surrogate</u>	<u>Recovery Limits %</u>
DCAA	37-140

4. Project specific limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

LABORATORY CONTROL STANDARD (LCS/LCSD)

1. A LCS and/or LCSD (if required) is included in each batch to demonstrate the system is in control. Routinely the LCS/LCSD will be spiked with the full list of analytes. The control limits are indicated on the following table:

TABLE I: LCS/LCSD, MS/MSD CONTROL LIMITS

COMPOUND	% RECOVERY WATER	RPD WATER	% RECOVERY SOLID	RPD SOLID
2,4-D	10-126	40	10-126	40
2,4,5-T	32-130	40	32-130	40
2,4,5-TP (Silvex)	34-124	40	34-124	40

All other compounds*	30-130	40	20-150	40
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*Included if full list spikes are required.

2. If a recovery is below the lower control limit, the batch must be re-extracted and re-analyzed. If a re-extraction is not possible due to insufficient sample volume, report the data with a narrative.
3. If the full list of target analytes is spiked, a small percentage of sporadic failures will be allowed. See Table 3 for the number of allowable failures. The failures are noted in the case narrative.

Table 3: Number of Allowable Failures

Number of Analytes	Failures Allowed
5-15	1
16-30	2

4. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

MATRIX SPIKES (MS/MSD)

1. The purpose of the MS/MSD is to assess the performance of the method for a particular sample matrix. The recoveries for each spike compound should be within the control limits specified in Table 2.
2. Whenever the MS and/or MSD recoveries are outside the control limits, review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
3. If recoveries for the MS and/or MSD are outside the control limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.
4. If precision is outside of control limits, re-extract the parent, MS, and MSD. If the failure is repeated, check that the samples are homogenous. Narrate the sample results including corrective action and sample appearance if applicable.
5. 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.
6. Spikes may be diluted out in the analysis process. Document this in the case narrative.
7. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in Table 2. See SOP GEN-019.

METHOD PERFORMANCE

- 1) In single laboratory studies using organic-free reagent water and clay/still bottom samples, the mean recoveries presented in Tables 4 and 5 of Method 8151A were obtained for diazomethane derivatization. The standard deviations of the percent recoveries of these measurements are also in Tables 4 and 5 of Method 8151A.
- 2) Table 6 of Method 8151A presents relative recoveries of the target analytes obtained using the PFBBR derivation procedure with spiked water samples.

POLLUTION PREVENTION

See QAPP Section 13.2

WASTE MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
WET LAB
STANDARD OPERATING PROCEDURE

PROCEDURES: WL-042
PAGE: 1 OF 17
EFFECTIVE DATE: 2/3/09
APPROVED BY: MAP
QA/QC APPROVED: JDT

SUBJECT SCOPE AND APPLICATION

This method addresses the sequential determination of the anions fluoride, chloride, bromide, nitrate, nitrite, chlorate and sulfate by ion chromatography. A water sample or combustate solution is injected into the eluent stream and passed through a series of ion exchangers. The ions of interest are separated on the basis of their relative affinities for a low capacity, strongly basic anion exchanger. The separated anions are directed through a hollow fiber cation exchanger membrane or micromembrane suppressor bathed in continuously flowing strongly acid solution (regenerant solution). In the suppressor the separated anions are converted to their highly conductive acid forms and the eluent is converted to weakly conductive carbonic acid. The separated anions in their acid forms are measured by conductivity. They are identified on the basis of retention time as compared to standards. Quantitation is by measurement of peak area.

MATRIX Water and Solid

REFERENCES EPA SW-846 Method 9056A
EPA 300.0

SAMPLE PRESERVATION Cool 4°C
Chlorate - Cool 4°C + 1mL EDA preservation solution to 1000 mL of sample

HOLDING TIME Bromide, Chloride, Sulfate, Fluoride - 28 days
Nitrate, Nitrite - 48 hours
Chlorate - 28 days

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety

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glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents and chemicals must be handled in ventilated hoods.

INTERFERENCES

Any substance that has the same retention time as a target anion and produces a detector response will interfere. This type of interference may be resolved by spiking the sample with the ion of interest to determine if the retention time is correct.

A high concentration of any one ion also interferes with the resolution, and sometimes retention, of other ions.

Sample dilutions can eliminate much interference. False peaks may result from contamination of the reagent water, glassware, or sample processing apparatus.

REAGENTS AND STANDARDS

Eluent - 20ml of Dionex AS14 eluent concentrate diluted to 2L in a volumetric flask with deionized water.

Stock Standards - 1000 mg/L Chloride, Fluoride, Sulfate, Bromide, Nitrate, Nitrite, Chlorate-Commercially prepared. Store standards in a cooler at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

A 100 mg/L anion working standard (mixed standards) is prepared from the 1000 mg/L stock standards. Transfer 10 mL of Fluoride, Chloride, Bromide, Sulfate, Nitrate and Nitrite to a 100 mL volumetric flask. Dilute to volume with DI water. Chlorate is prepared as a single analyte by transferring 0.1 mL of Chlorate stock (1000 mg/L) to a 20 mL volumetric flask and diluting to volume with DI Water.

Calibration (Mixed Anions)

NOTE: Before analyzing standards, analyst should add 100 μL of Eluent concentrate to 10 mL of the standard.

- Level 1 (Blank): DI Water
- Level 2 (0.1 mg/L): is prepared by transferring 0.1 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric flask and diluting to volume with DI water.
- Level 3 (0.2 mg/L): is prepared by transferring 0.2 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric

- flask and diluting to volume with DI water.
- Level 4 (0.5 mg/L): is prepared by transferring 0.5 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric flask and diluting to volume with DI water.
- Level 5 (1.0 mg/L): is prepared by transferring 1.0 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric flask and diluting to volume with DI water.
- Level 6 (2.0 mg/L): is prepared by transferring 2.0 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric flask and diluting to volume with DI water.
- Level 7 (5.0 mg/L): is prepared by transferring 5.0 mL of the 100 mg/L mixed anion working standard to a 100 mL volumetric flask and diluting to volume with DI water.

Calibration (Chlorate Standard)

- LEVEL 6 (20mg/L): is prepared by transferring 2.0 mL of Chlorate standard (100 mg/L) to a 100 mL volumetric flask and diluting to volume with DI water.
- LEVEL 5 (10 mg/L): is prepared by transferring 50 mL of LEVEL 6 to a 100 mL volumetric flask and diluting to volume with DI water.
- LEVEL 4 (5.0 mg/L): is prepared by transferring 50 mL of level 5 to a 100 mL volumetric flask and diluting to volume with DI water.
- LEVEL 3 (2.5 mg/L): is prepared by transferring 10 mL of LEVEL 6 to a 100 mL volumetric flask and diluting to volume with DI water.
- LEVEL 2 (1.0 mg/L): is prepared by transferring 10 mL of LEVEL 5 to a 100 mL volumetric flask and diluting to volume with DI water.
- LEVEL 1 is a blank: DI water

Calibration Concentrations

	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluoride	0.1	0.2	0.5	1.0	2.0	5.0
Chloride	0.1	0.2	0.5	1.0	2.0	5.0
Bromide	--	0.2	0.5	1.0	2.0	5.0
Sulfate	--	0.2	0.5	1.0	2.0	5.0
Nitrate	--	0.2	0.5	1.0	2.0	5.0
Nitrite	--	0.2	0.5	1.0	2.0	5.0
Chlorate	1.0	2.0	5.0	10.0	20.0	--

Continuing Calibration Standard (CCV) - Prepare a mixed standard at 2.5 mg/L by adding 2.5 mL of the 100 mg/L mixed anions working standard to a 100 mL volumetric flask and diluting to volume with deionized water. Prepare Chlorate at the Level 5 concentration.

Independent Mixed Anion Stock Standard (1000/2000 mg/L) - Commercially prepared. This standard contains Chloride and Fluoride at 1000 mg/L and Bromide and Sulfide at 2000 mg/L.

Independent Nitrate Stock Standard (1000 mg/L) - Commercially prepared.

Independent Nitrite Stock Standard (1000 mg/L) - Commercially prepared.

ICV/LCS (1.5/3.0 mg/L) - Prepare a mixed anion working standard by adding 150 uL of the Independent mixed anion stock standard and 300 uL each of the Independent Nitrate and Nitrite stock standards to a 100 mL volumetric flask and dilute to volume with DI water. This will yield a concentration of 1.5 mg/L for Chloride and Fluoride and a concentration of 3.0 mg/L for Bromide, Sulfate, Nitrate, and Nitrite.

LLC 1 (0.1 mg/L) - Prepare the same as the Level 2 from the calibration curve. This standard is for Chloride and Fluoride only.

LLC 2 (0.2 mg/L) - Prepare the same as the Level 3 from the calibration curve. This standard is for Bromide, Sulfate, Nitrate, and Nitrite only.

LCR 1 (0.5 mg/L) - Prepare the same as the Level 4 from the calibration curve.

LCR 2 (2.0 mg/L) - Prepare the same as the Level 6 from the calibration curve.

LCR 3 (5.0 mg/L) - Prepare the same as the Level 7 from the calibration curve.

LCR B (0 mg/L) - Prepare the same as the Level 1 from the calibration curve.

Dionex AS 14 Eluent Concentrate

DI Water

APPARATUS

Dionex Series 500i Ion Chromatograph with eluent de-gas module, GP40 gradient pump (pump volume 1.2 mL/min), and ED40 electrochemical detector, 4mm/2 coil 0.5-1.5 mL/min sample loop, with computer and peaknet software.

Dionex Automated Sampler

Dionex AS14 and AG14 columns

Poly autosampler 5.0ml vials and filter caps
Lachat

Eluent reservoir

ASRS Ultra 11 4-mm suppressor

Helium (5.0 grade) cylinder regulated at 75 psi -
instrument setting 10 psi

Volumetric flasks

Mechanical pipette

Disposal pipettes

Specimen cups
Mechanical shaker
Analytical balance, calibrated each day of use

PROCEDURE

INSTRUMENT SETUP

1. Turn instrument on (only if power is off). Verify the gas regulator on the instrument is set at 10 psi.
2. Prepare schedule for run. Follow instructions included in the initial calibration or sample analysis sections.
3. Go to the peaknet main menu: select Run
4. Go to File: Load Method: Select method indicated in schedule: Open
5. The pump will turn on and eluent will begin flowing through the column.
6. Run a blank to stabilize instrument.

RETENTION TIME WINDOWS

Take at least three measurements over at least a 24-hour period. The retention time window is \pm three times the standard deviation over the measurements. Retention Time Windows will be evaluated annually or whenever major instrument maintenance is performed.

INITIAL CALIBRATION

1. Prepare a calibration curve by analyzing each calibration standard. Pour the standards into autosampler vials and load on the autosampler in the assigned positions.
2. From the peaknet main menu: select the schedule button.
3. Double click on the box in the first row of the method column. Select DX500.met (desired method)

4. Click on the first row beneath data file. Type in the date (month and day-ex. 0116). Cursor down 16 positions. The method data file column should copy itself over and over.
5. In the sample column, type IB, Autocal1R, Autocal 2R, Autocal3R, Autocal4R, Autocal5R, Autocal6R, Autocal7R, ICV, ICB, LLC1, LLC2, LCRB, LCR1, LCR2, LCR3, and Stop.
 - A. In the sample type column, the first two should be sample.
 - B. Beside autocal1R, click on the down drop arrow and select calibration standard. In the level, choose the correct level starting with 1.
 - C. Repeat B for each calibration level.
 - D. For each position following Autocal7R, leave the sample type as sample.
 - E. Beside stop, double click on the method and select "shutdown.met".
6. Start the calibration.
7. After the calibration is completed, select method from the main menu. Click on file, then open.
8. Double click on desired calibration method.
9. Under data processing, click on the component table (5th from left).
10. The list of components will be on the screen. Click on details to see the plot and area count of that component.
11. Click on arrow in top left to see next component. The peaknet software will calculate the correlation coefficient. The correlation coefficient must be 0.995 or greater. If the correlation coefficient is not acceptable, check peaks for identification, proper integration, and area count update. If no problem can be identified, reanalyze the calibration. If a problem is identified with one standard, it is acceptable to rerun that one level. Print the plot if the correlation is acceptable.

Quadratic curve fits may not be used.

12. Check the %residuals on each level of each analyte. All % residuals must be less than 10% except for the low level for each analyte. The low level must be less than 25%. See calculation section for equation to calculate % residual. If residuals fail check integration. If the %residuals still fail, a new calibration must be performed.
13. Analyze an independent standard immediately after calibration as a second source verification of the calibration standards. If the second source verification falls outside of the 90-110% control limits, verify identification of each analyte and check integrations. If a problem is not identified, recalibrate.
14. Analyze an ICB (calibration blank). Results must be less than $\frac{1}{2}$ the reporting limit. If it is greater than this, determine the source of the contamination, perform corrective action, and repeat.
15. Analyze low-level check standards, prepared at the initial calibration Level 2 for Chloride and Fluoride and at the Level 3 calibration for the remaining analytes. Determine recovery. Acceptance limits are 50-150%. Some programs may require more stringent criteria. Check SOP GEN-019. If the criterion is not met determine the source of the problem and repeat. If the recovery continues outside of criterion, recalibrate.
16. Linear Calibration Range Verification - Following calibration, the linear range of the curve must be verified. Analyze LCR standards and the LCR blank. Determine the recovery. Acceptance limits for all analytes are 90-110%. The blank must be less than $\frac{1}{2}$ the reporting limit. If any analyte fails the criteria, remake the standard and reanalyze. If it still fails, recalibrate. This procedure must be performed for all new calibrations and then again 6 months if the curve is still being used. All LCR data should be filed with the corresponding calibration curve.

17. A new calibration must be performed if the CCV criteria is not met, when the calibration stock standards expire or at least annually.

CONTINUING CALIBRATION VERIFICATION (CCV)

1. An existing curve (stored) is verified by analyzing a CCV. If the percent recovery is 90 - 110% and the retention time is +/-10% of the original calibration curve, the analysis can continue. Check that each analyte meets the above criteria. The CCV's will be included in the sequence prepared for sample analysis. If the percent recoveries or the retention times are still outside the control limits, prepare a new standard and reanalyze. If the percent recoveries or the retention times are still outside the limits, a new calibration curve must be performed.
2. After the initial CCV, a CCV is analyzed after every 10 injections and at the end of the run. The recovery must be 90-100% and the retention time of each analyte must be within the calculated retention time window of the initial CCV for the day. Reportable samples and QC must be bracketed by acceptable CCV's. Rerun any samples or QC that are not bracketed by acceptable CCV's.
3. Each CCV will be followed by a CCB (blank - deionized water). The concentration should be less than $\frac{1}{2}$ the reporting limit for each target analyte. If a target analyte is detected above this limit, the data may only be reported if the analyte is not detected in the associated sample(s) or if the CCB concentration is < 5% of the measured concentration in the associated sample(s).
4. Prepare the CCV/CCBs and pour into autosampler vials and load into an autosampler tray. The positions are identified in the sample sequence.
5. Project specific requirements may apply for CCV and CCB and may be more stringent. See SOP GEN-019.

DOWNLOADING A SCHEDULE

1. Build the sample batch in Horizon.
6. Log-on to the Horizon Report Manager using the LIMS login ID and password.
7. Select Workflow. Under topics subheading, select Worklists and downloads. Under actions, select Dionex download and click "OK".
8. Select a Queue: Arrow down and highlight "IC".
9. A sequence number will automatically be generated and appear on the screen. Batch number is the horizon batch number. Enter the number by hitting the enter key. A batch number will appear on the right. In the instrument field, type IC1 and hit the enter key.
10. Select "OK" in the box that appears on the screen.
11. Return to the peaknet main menu and select schedule.
12. Select File: Import ASCII file.
13. A box with "Select a text file to import into current schedule" will appear. Use the schedule drop down menu and select: pcommon on "GCAL1" (N:): Arrow down to Dionex and double click. Go to the files of type: click on the down drop menu and choose "all files". Double click on "IC1". Choose the date you download from Horizon Report Manager and double click on it. The horizon batch numbers that are available will show on the screen. Choose the batch that will be analyzed by double clicking on the number.
14. Sample numbers will now show up in the schedule.
15. Find the row with the heading Data File. Type in the month and day (ex: 0116) in each block of each line of schedule.
16. Save file as month, day, and number of schedule. (ex: 0116-1). Exit and return to the peaknet main

menu.

17. Click on "Run". The pump will turn on.
18. Click on file and highlight "Load Schedule" and click. In the box next to file name, type the file name (ex: 0116) or arrow over and choose schedule by double clicking on it. Click on "OK".
19. Use the schedule to load samples into the correct autosampler positions.

MANUALLY TYPING A SCHEDULE

1. Build BSWA and print.
2. From Peaknet main menu, select "SCHEDULE".
3. Double click on the first box below the word 'Method'. Select the method of choice by highlighting the method and clicking open.
4. In the box to the right of the method is the Data File. Type in the month and day (ie. 1110 for November 10th).
5. Go to the first box beneath the sample, this should be the IB (instrument blank).
6. Arrow down to line 2. This block should have CCV in the field.
7. Arrow down to line 3. This should have CCB in the field.
8. Arrow down until all of the QC and samples on the BSWA are listed in the schedule.
9. Insert a CCV and CCB after every tenth position.
10. Continue with steps 16 - 19 under "Downloading Schedule" described above.

SAMPLE PREPARATION

1. Samples may be screened using the Lachat, especially if high chloride concentrations are suspected. See SOP WL-038 for procedure.
2. Waters - To 10 mL of each sample, add 100 uL of Eluent concentrate. Pour each sample into an autosampler vial, cap, and load on the autosampler tray in the assigned position.
3. Solids -
 - A. Soils - Weigh 10 g of sample in a specimen cup.

Add 100 mL of deionized water. Place in the mechanical shaker for at least 2 hours. Allow extract to settle for a minimum of 12 hours. Use a 0.45um filter to filter the extract before analyzing on the instrument. Pour the filtered extract into an autosampler vial and load on the autosampler tray in the assigned positions.

- B. Liquid organic samples - 1 gram of sample is combusted with Parr bomb apparatus, diluted with eluent to 100ml, and collected in a specimen cup. Pour the combustate into an autosampler vial and load on the autosampler in the assigned positions. Refer to heat of combustion SOP for combustion procedure. Enter the sample prep into the heat logbook (example page attached). Enter a comment into the logbook if cleaning was necessary following the combustion.

SAMPLE ANALYSIS

1. Allow the system to run for approximately 3-5 minutes before starting the run. Run a blank to stabilize the instrument at the start of each day. This blank is not evaluated.
2. Load the autosampler trays into the autosampler. Press Hold/Run. A green light will be on the correct run.
3. From the peaknet run-Gradient menu go to "run" and click on start. The instrument will begin running. Minimize the screen. "DO NOT CLOSE".
4. Throughout the run, check for signs of an unstable baseline.
5. As samples are completed, a data file will print, which contains the chromatograph and the analyte concentrations. The analytes are identified by peaks that fall within the defined window.
6. Samples with concentrations greater than the highest standard should be diluted and reanalyzed.
7. Check that retention times for each ion is correct. If the resulting chromatogram fails to produce adequate

resolution or if the identification of the anions is questionable, spike the sample and reanalyze.

8. Download the data to the LIMS. Complete the bench sheet as shown on the attached example.

CALCULATIONS

Water mg/L = Instrument reading X DF

DF = Dilution Factor

% Residual = $\frac{\text{Calculated Amount}}{\text{True Amount}} * 100$

NOTE: If the dilution is entered in the schedule, the instrument reading will be dilution corrected.

Solid mg/kg = $\frac{\text{Instrument reading} \times \text{FV} \times \text{DF}}{\text{Sample Wt (g)}}$

FV = Final Volume of the Combustate or Extract

Total Halides is the sum of Cl, F, and Br

Total Organic Halides is the sum Cl, F, and Br for both soils and liquid organic.

NOTE: The LIMS will calculate the final results for solids. After downloading the dilution corrected concentration, the LIMS will multiply by the final volume and divide by the initial weight.

Linear Calibration Calculations for Least Square Regression:

1. $y = mx + b$

where y = area of analyte
 m = slope of line
 x = concentration of analyte
 b = the y intercept

2. $m = \frac{n\sum xy - \sum x \sum y}{n\sum x^2 - (\sum x)^2}$

where n = number of calibration standards

$$3. \quad b = \bar{y} - (m \bar{x})$$

where \bar{y} = average area in calibration

\bar{x} = average concentration in calibration

$$4. \quad \text{Correlation Coefficient (r)}$$

$$r = \frac{n \sum xy - \sum x \sum y}{(\sqrt{n \sum x^2 - (\sum x)^2}) (\sqrt{n \sum y^2 - (\sum y)^2})}$$

$$5. \quad \text{Coefficient of Determination (r}^2\text{)} = r \times r$$

QUALITY CONTROL

INDEPENDENT CALIBRATION VERIFICATION

1. Analyze the ICV after initial calibration. The recovery must be 90-110% for the calibration to be accepted. If unacceptable, remake and re-analyze one time. If the recovery is still unacceptable, determine if there is a problem with either standard stock solution. Recalibrate if it is determined that the problem is the calibration standards.

CCV and CCB

1. Refer to the continuing calibration verification section of the SOP.

METHOD BLANK

1. Analyze one method blank with each batch of twenty or fewer samples. No target analytes should be detected in the method blank above $\frac{1}{2}$ the reporting limit. If any target analytes are detected, data may not be reported and must be reanalyzed unless the following apply.
 - A. If a target analyte is detected above $\frac{1}{2}$ the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
 - B. If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.

LABORATORY CONTROL STANDARD (LCS)

1. Analyze one LCS with each batch of twenty or fewer samples. The recovery must be 90-110%. If a recovery is high with no hits in the associated samples, the data may be reported with a narrative. A LCSD may be analyzed if required for specific projects. The LCS/LCSD RPD should be $\leq 15\%$.
2. A combustate LCS prepared from an oil standard is included with each batch of twenty or fewer samples prepared by method 5050. The control limits are 80-120%. If the LCS recovery is outside the control limits, the samples must be re-prepped and reanalyzed.

MATRIX DUPLICATES

1. Duplicate one sample in each batch of twenty or fewer samples of each matrix type. Duplicate analyses should have an RPD of ≤ 15 . If duplicate analyses are outside of the RPD control limits, the duplicate must be reanalyzed unless the concentration of the original sample and duplicate are $< 5 \times$ the reporting limit. If this is the case, the data can be reported as long as a narrative is included in the report.

MATRIX SPIKES

1. Perform a matrix spike in each batch of 20 or fewer samples for each matrix type. The recovery should be 80-120%. A MSD may be required for specific projects. The MS/MSD RPD should be ≤ 15 . If the MS recovery falls outside of the laboratory control limits, an MSD must be performed. If the MS/MSD RPD meets the criteria of 25%, the data is reportable as long as a narrative is included in the report.

NOTE: For Method EPA 300.0, perform a matrix duplicate and a matrix spike in each batch of 10 or fewer samples.

PROJECT SPECIFIC LIMITS

Project limits may apply and may be more stringent. See SOP GEN-019.

REPORTING LIMITS

The following table lists default reporting limits. Reporting limits may be lowered, when allowable, by calibrating lower. The reporting limit should be no lower than 2X the verified MDL. Some projects may have different criteria for setting reporting limits. Check SOP GEN-019 and with the project manager.

<u>ANALYTE</u>	<u>WATER</u> <u>mg/L</u>	<u>SOLID</u> <u>mg/kg</u>	<u>SOLID COMBUSTATE</u> <u>mg/kg</u>
Fluoride	0.1	10	100
Chloride	0.1	10	100
Bromide	0.2	20	200
Sulfate	0.2	20	200
Chlorate	1.0	NA	NA
Total Halides	NA	4.0	40.0
Nitrate	0.2	2.0	NA
Nitrite	0.2	2.0	NA

MANUAL
INTEGRATIONS

Perform and document manual integrations as described in SOP QA-010.

METHOD
PERFORMANCE

- 1) Single-operator accuracy and precision for reagent, drinking and surface water, and mixed domestic and industrial wastewater are listed in Table 3 of Method 9056.
- 2) *Combustate samples* - These data are based on 41 data points obtained by six laboratories that each analyzed four used crankcase oils and three fuel oil blends with crankcase in duplicate. The oil samples were combusted using Method 5050. A data point represents one duplicate analysis of a sample. One data point was judged to be an outlier and was not included in the results.
 - a) *Precision* - The precision of the method as determined by the statistical examination of interlaboratory test results is as follows:

Repeatability - The difference between successive results obtained by the sample operator with the same apparatus under constant operating conditions on identical test material would exceed, in the long run, in the normal and correct operation of the test method, the following values only in 1 case in 20. (See table 4 of Method 9056)

x = the average of two results in ug/g.

$$\text{Repeatability} = 20.9 \sqrt{x^*}$$

Reproducibility - The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would exceed, in the long run, the following values only in 1 case in 20:

$$\text{Reproducibility} = 42.1 \sqrt{x^*}$$

x = the average of two results in ug/g.

b) *Bias* - The bias of this method varies with concentration, as shown in Table 5 of Method 9056.

$$\text{Bias} = \text{Amount found} - \text{Amount expected}$$

POLLUTION PREVENTION	See QAPP Section 13.2
WASTE MANAGEMENT	See SOP GEN-009
IDOC	See SOP QA-014
MDL	See SOP QA-009

GULF COAST ANALYTICAL LABORATORIES, INC
METALS
STANDARD OPERATING PROCEDURE

PROCEDURE: MET-010
PAGE: 1 of 16
EFFECTIVE DATE: 04/09/09
APPROVED BY: *MAT*
QA/QC APPROVED: *JBT*

SUBJECT SCOPE AND APPLICATION

This method covers the operation of the Perkin Elmer 5300DV and 4300DV ICPs. The procedure is applicable for the analysis of aqueous samples, TCLP extracts, EPTOX extracts, soils, solids, and domestic or industrial wastes for dissolved or total metals. The sample is analyzed after preparation by the appropriate EPA, SW846, NIOSH, or ASTM procedure. Elements currently analyzed using this method are listed in Table I.

MATRIX Water and Solid

REFERENCES EPA (EMMC) 200.7 - Determination of Metals and Trace Metals in Water and Wastes by ICP-Atomic Emission Spectrometry, Revision 4.4 (EPA Region VI Approval) (A)
200.7 - CFR 40 Part 136, Appendix C, "Inductively Coupled Plasma Atomic Emission Spectrophotometric for Trace Element Analysis of Water and Wastes" (If Required) (B)
SW846 6010C - for South Carolina and North Carolina Projects
SW846 6010B
Perkin Elmer Instrument Manuals
WinLab Software Guide

HOLDING TIME & PRESERVATION

Dissolved Metals (waters)- Filtered through a 0.45 um membrane filter then HNO₃ to pH < 2; Digestion required for 200.7 (SOP MET-005)-6 months

Total Metals (waters)- HNO₃ to pH < 2; Digestion required (SOP MET-005)-6 months

Total Metals (solids) - Cool 4°C; Digestion required (SOP MET-004, MET-018)-6 months

DEFINITIONS See SOP GEN-016

SAFETY

1. All employees will view safety video orientation prior to performing work within the laboratory.
2. Whenever any acids are used, safety glasses must be worn. When working with acids, a face shield and an acid resistant apron should be worn. Acid resistant gloves should be worn at all times.
3. Evaporation and/or digestion with acids must be

performed under a well-ventilated acid resistant fume hood.

4. Each chemical compound must be treated as a potential hazard. Exposure to these chemicals must be reduced to the lowest possible level by means of fume hoods, respirators and protective clothing.
5. A reference file of material safety data sheets (MSDS) are available to all personnel to provide knowledge on safe handling of chemicals used in this procedure.

INTERFERENCES

Several interferences are possible. These include -

1. Spectral Interferences can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line.

Automatic interference correction for condition (1) above is done by the instrument software. The analyst does not have to manually do the correction. Background correction intervals to correct conditions (3) and (4) have likewise been automatically set up in the instrument software.

2. Physical Interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties 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, using a peristaltic pump and/or using an internal standard element.
3. Chemical Interferences are characterized by molecular

compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

APPARATUS

Perkin Elmer 5300DV/4300DV Inductively Coupled Plasma with WinLab 32 software
Perkin Elmer As 90 Autosampler
Peristaltic pump and tubing
Autosampler tubes
Mechanical pipets and pipet tips
Volumetric Flasks - Class A
Argon and compressed air supply with pressure controls

REAGENTS
AND STANDARDS

1. Deionized water - monitored daily.
2. Concentrated nitric acid - trace metals grade, label with date opened and enter into reagent logbook
3. Concentrated hydrochloric acid - trace metals grade, label with date opened and enter into reagent logbook
4. 5% Nitric Acid - prepared from the trace metals grade concentrate
5. 6% Nitric Acid/10% Hydrochloric Acid - Calibration Blank
6. Stock standard metals solutions (1000 mg/L) - commercially prepared, label with date opened.
7. Custom mixed standards - commercially prepared, label with date opened.
8. Calibration standards: A custom made solution for calibrations standards is used. See Table I for the elements included and concentration. Prepare all standards in the 6% Nitric Acid/10% Hydrochloric Acid.
9. Continuing Calibration Verification (CCV) standard - is prepared using the calibration standard. The CCV

includes equal aliquots of the calibration standard and 6% Nitric Acid/10% Hydrochloric Acid.

10. Second Source Stock Standards - Two multi-element custom solutions-commercially prepared.
11. Initial Calibration Verification (ICV) standard - Dilute the appropriate amount of the second source multi-element stock solution to volume with 6% Nitric Acid/10% Hydrochloric Acid. The concentration of the elements in the stock solutions vary in concentration, therefore the analyst should prepare this solution such that the majority of the elements are in the middle of the calibration range.
12. Interference solution stock standards - available commercially.
13. Interferent Calibration Solution A (ICSA) - Pipet 8.0 mL of the interferent stock Spex Int. A-1 into a 200 mL volumetric flask and dilute to volume with 6% Nitric Acid/10% Hydrochloric Acid. Final concentrations are: Al, Ca, Mg - 200 mg/L; Fe - 80 mg/L.
14. Interferent Calibration Solution B (ICSAB) - Pipet 8.00 mL of the interferent stock Spex Int. A-1, 2.0 mL of interferent stock Spex Int. B-1, and 2 mL of interferent stock Spex Int. B-2 standards into a 200 mL volumetric flask and dilute to volume with 3% Nitric Acid/6% Hydrochloric Acid. Final concentrations of interferents are as in (13) above; concentrations of minor elements are either 0.500 or 1.00 mg/L.
15. Internal Standard - 5 mg/L Yttrium, 5 mg/L Scandium.
16. Low-level Calibration Verification Standard Composed of two custom made standards prepared at the reporting limit.
17. All solutions are prepared in labeled, dedicated glassware. The glassware is rinsed in an acid solution to clean.
18. All reagents used must be entered in the reagent logbook, except for water, which is monitored by the QA/QC Department. Standards prepped are entered into the standard prep logbook.

INSTRUMENT
SET-UP

1. Check Argon and compressed air supply.
2. Check wash solution in autosampler flush container. Fill if necessary (6% Nitric Acid, 10% Hydrochloric Acid).
3. Check drain bottle below instrument. Empty if necessary.
4. Connect the peristaltic pump tubing between the posts on the pump (Sample pump and internal standard pump). Close the platens.
5. Restart the computer. Select the Optima ICON on the Windows Desktop. Select a workspace. Select a method.
6. Select the Plasma ICON. Select "ON" button. Allow Plasma to warm up 45 minutes.
7. Set up bench sheet by filling in the positions with the samples to be analyzed. Include the standard identification numbers for the calibration standards, ICV, ICSA, and ICSAB on the bench sheet (Attachment 1).
8. Load the autosampler by filling the tubes (10 mL will suffice) and inserting them into the positions corresponding to those on the bench sheet. Verify all blank, standard and QC sample tubes are filled.
9. Open a sample information file on the set up window and enter the analytical sequence. <SAVE>. Open a Results file - name by date (YYMMDD).
10. If the torch compartment has been opened, go to Tools pull down menu. Select Spectrometer Control. Aspirate a 5mg/L Y solution. Select Optimize X-Y. Select Hg Realign. Record the Hg intensity in the maintenance log.
11. Go to analyze in the Analysis Control Window.

CALIBRATION AND
SAMPLE ANALYSIS

1. Select "Analyze All". Instrument will calibrate and continue with QC and sample analysis. The calibration

is at one point with a blank. It is verified with an ICV, LLICV, CCV and CCB. Once data is collected, and instrument QC is evaluated, the data can be posted. This is performed by direct upload of data using LIMSLink Software.

2. SHUT DOWN - If an additional run will not be performed select auto-shutdown from the system pull down menu.
3. Samples are diluted if above linear range as described in QC section 12 of this SOP.
4. Replicates are evaluated for all runs. If the RSD is more than 5%, each replicate is evaluated. If an outlier is determined, it may be excluded. This is documented on the metals run log and in the raw data.

REPORTING
LIMITS

The following charts indicate reporting limits for EPA 200.7, SW-846 6010, TCLP extracts, SPLP extracts and UTS analyses:

EPA 200.7:

ANALYTE	WATER (mg/L)
Aluminum	0.2
Antimony	0.06
Arsenic	0.04
Barium	0.01
Beryllium	0.005
Boron	1
Cadmium	0.005
Calcium	0.05
Chromium	0.01
Cobalt	0.01
Copper	0.01
Iron	0.05
Lead	0.015
Lithium	0.05
Magnesium	0.05
Manganese	0.015
Molybdenum	0.03
Nickel	0.04
Potassium	0.2
Selenium	0.04
Silver	0.01
Sodium	1
Strontium	0.01
Thallium	0.02

Tin	0.025
Titanium	0.1
Vanadium	0.02
Zinc	0.02
Zirconium	0.01

SW-846, 6010:

ANALYTE	WATER (mg/L)	SOLID (mg/kg)
Aluminum	0.2	8
Antimony	0.06	2.4
Arsenic	0.04	1.6
Barium	0.01	0.4
Beryllium	0.005	0.2
Boron	1	40
Cadmium	0.005	0.2
Calcium	0.05	2
Chromium	0.01	0.4
Cobalt	0.01	0.4
Copper	0.01	0.4
Iron	0.05	2
Lead	0.015	0.6
Lithium	0.05	2
Magnesium	0.05	2
Manganese	0.015	0.6
Molybdenum	0.03	1.2
Nickel	0.04	1.6
Potassium	0.2	8
Selenium	0.04	1.6
Silver	0.01	0.4
Sodium	1	40
Strontium	0.01	0.4
Thallium	0.02	0.8
Tin	0.025	4
Titanium	0.1	4
Vanadium	0.02	0.8
Zinc	0.02	0.8
Zirconium	0.01	0.4

TCLP, UTS, SPLP Extracts:

ANALYTE	TCLP (mg/L)	UTS (mg/L)	SPLP (mg/L)
Aluminum	----	----	0.2
Antimony	0.06	0.1	0.06
Arsenic	0.2	0.14	0.04

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Barium	1	1	0.01
Beryllium	0.005	0.01	0.005
Boron	----	----	1
Cadmium	0.01	0.01	0.005
Calcium	----	----	0.05
Chromium	0.05	0.05	0.01
Cobalt	----	----	0.01
Copper	----	----	0.01
Iron	----	----	0.05
Lead	0.1	0.075	0.015
Lithium	----	----	0.05
Magnesium	----	----	0.05
Manganese	----	----	0.015
Molybdenum	----	----	0.03
Nickel	0.04	0.05	0.04
Potassium	----	----	0.2
Selenium	0.1	0.2	0.04
Silver	0.05	0.01	0.01
Sodium	----	----	----
Strontium	----	----	0.01
Thallium	0.02	0.02	0.02
Tin	----	----	0.025
Titanium	----	----	0.1
Vanadium	0.02	0.02	0.02
Zinc	0.1	0.5	0.02
Zirconium	----	----	0.01

NOTE: Reporting limits may vary according to specific project requirements.

QUALITY CONTROL &
DATA ACCEPTANCE
CRITERIA

1. The Initial Calibration Verification (ICV) standard recoveries must be 95-105% for 200.7 and 90-110% for 6010. This is performed daily following the initial calibration. The ICV standard should be prepared from an independent (second source) material at or near the mid-range of the calibration curve.
2. Analyze a Initial Calibration Blank (ICB) to verify all concentrations are below the reporting limit. The concentration must be $\leq \pm$ the reporting limit. Additional requirements may apply and may be more stringent. See SOP GEN-019.

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3. Analyze a Low-level Initial Calibration verification (LLICV) prepared at or below the project required reporting limit. The recovery for this check standard must be 70-130% for 6010C or 50-150% for 6010B. Project specific limits may be required, see SOP GEN-019.
4. The ICSA solution is used to monitor the interelement correction factors for the common interferences (Fe, Ca, Mg, Al). This solution is not required in the methods but will be used to evaluate elements that are not spiked in the ICSAB. The concentration for these elements must be $\leq \pm$ the reporting limit. The ICSA concentration must be less than or equal the reporting limit and greater than or equal to -2 times the reporting limit. Additional requirements may apply and may be more stringent. See SOP GEN-019.
5. All elements in the ICSAB solution should have recoveries of 80-120%. Results falling outside these limits may indicate the need for re-generation of the inter-element correction factors (IECs).
6. Continuing Calibration Verification standards (CCV), Continuing Calibration Blanks (CCB), and for 6010C Low-level Continuing Calibration Verification (LLCCV) are run every 10 samples and at the end of the analytical run. The first CCV recovery must be 95-105% and all other CCV recoveries for the analytical run must be 90-110% for 200.7. For 6010B and 6010C, the CCV recoveries for all target analytes -90-110% for 6010. For 6010C, the recovery for the LLCCV must be 70-130% for all analytes. The RSD between the replicates should not be greater than 5%. If this is exceeded evaluate the to determine if re-analysis is necessary. The CCB concentration should never exceed the reporting limit. If a CCV and/or CCB is outside control limits, re-analyze one time (additional runs are acceptable if a documented reason for the failure is available) and if the values are still outside the control limits, recalibrate and re-run the previous 10 samples. Results are reportable with a narrative if the CCB fails and there are no hits in the associated samples. Additional requirements may apply and may be more stringent. See SOP GEN-019.
7. Method Blank - one method blank is prepped with each batch of 20 or fewer samples. The concentration of each element should never exceed the reporting limit for all

associated samples. Detected levels of elements in the method blank must be less than 10% of the regulatory limit or the sample concentrations for the data to be reported. If the data is reported make a note of the problem in the case narrative. Find the source of the problem and correct and re-analyze if possible. Additional requirements may apply and may be more stringent. See SOP GEN-019.

8. A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD-if required for project or if insufficient sample if available for a matrix duplicate or MS/MSD) is prepped with each batch of 20 or fewer samples. The recovery must be 80 - 120% for 6010B&C waters and solids, 85-115% for 200.7, and 70-130% for 6010B&C organics. The LCS/LCSD RPD should be $\leq 20\%$. If the LCS recovery is outside the control limits, the sample batch must be re-prepped. Samples may be reported if the LCS fails high with no hits in associated samples. Report with a narrative describing the failure.
9. The relative percent difference (RPD) for matrix duplicates must be $\leq 20\%$, unless the sample concentration is less than five times the detection limit or the sample matrix is determined to be heterogeneous. Additional requirements may apply and may be more stringent. See SOP GEN-019.
10. Matrix spike (MS) and Matrix Spike Duplicate (MSD) recoveries must be 75 - 125% for 6010B&C water and solids, 70-130% for 200.7 and 6010B&C organics. The MS/MSD RPD should be $\leq 20\%$. MSD's are prepped and analyzed if there is sufficient sample. If the recovery is outside these limits, the data should be flagged. A spike is not applicable if the sample concentration is greater than 4 times the spike level. Interference checks shall be performed for samples with spike recoveries outside the control limits. If a TCLP extract spike recovery for a regulated metal is $<50\%$ and the sample concentration is within 20% of the regulatory level the sample must be reanalyzed by the method of standard additions. Additional requirements may apply and may be more stringent. See SOP GEN-019.
11. For solid samples that require Antimony analysis - If the MS and MSD recovery for Antimony are outside of the control limits of 75-125%, the data should be flagged. In

addition, if the recovery for Antimony is <10%, the samples should be reprepared for Antimony only, using the optional procedure for Antimony found in SOP MET-004. Also, if historical data for a particular sample indicates low recoveries for Antimony, the analyst should proceed directly to the optional steps.

12. Interference checks:

A. Dilution Test - Perform a 5x dilution on one sample per batch. The concentrations of the elements in the sample to be diluted should be greater than 50 times the MDL. The neat sample concentration and five times the diluted sample concentration should agree within 10%. If not, a chemical or physical interference should be suspected.

B. Post-Digestion Spike - Analyte is added to a portion of a digested sample, or its dilutions. A post-digestion spike is also performed on all samples with MS/MSD recoveries outside control limits. If the recovery is outside control limits of 80-120% for 6010C or 75-125% for 6010B, all data for the batch will be flagged by including a narrative with the data. Internal standardization is utilized therefore the method of standard additions is not required. A post-digestion spike is required for each sample batch. It is usually performed on the sample chosen as the matrix spike.

13. Samples with interferent or analyte concentrations greater than the determined instrument linear range will be diluted and reanalyzed. Target analytes must always be diluted to within the linear range of the instrument. Samples must be diluted for non-target analytes only if that analyte is an interferent for a target analyte. Dilutions for interferents above the linear range should only be performed if a correction factor is in effect for target analytes. Do not over dilute. Perform the minimum dilution possible to dilute within the linear range of the instrument for any analyte. Additional requirements may apply and may be more stringent. See SOP GEN-019.
14. Review the Internal standard recovery for each sample. Perkin Elmer recommends recovery limits of 50-150%. Review the data thoroughly before accepting or rejecting data based on these control limits. Determine if high or

low recoveries are due to instrument drift by reviewing the internal standard recoveries for CCV's and CCB's. This will eliminate performing unnecessary dilutions if the sample is reanalyzed. High recoveries may be the result of a clogged sample uptake tube or an empty sample vial. Extremely high recoveries can result from the presence of the internal standard element in the original sample. If this is suspected, reanalyze the sample without the internal standard, or re-analyze using a different IS such as scandium. If the analyst determines the recovery is outside the control limits due to a matrix interference, then the sample must be reanalyzed diluted.

15. Determine IEC's semi-annually.
16. Determine linear ranges semi-annually. Spike at historical values. Each metal must be recovered at 90-110%.
17. Determine MDL's annually using the requirements of SOP QA-009. MDL's shall be determined more frequently if there is a change to the method or instrumentation that may alter the sensitivity of the measurement. An MDL study is performed for each instrument. MDL's should be less than $\frac{1}{4}$ the reporting limit, but cannot be higher than the reporting limit. The MDL's are confirmed by the analysis of a lower limit of quantitation check sample (MDL check). The MDL check is prepared like a sample and spiked at the concentration of the LLICV. The analytes must be detected within +/- 30% of the true value.
18. Determine IDLs every three months. Additional requirements may apply and may be more stringent. See SOP GEN-019.

MAINTENANCE

1. Clean the nebulizer periodically in a 5% nitric acid solution. Sonication of the nebulizer in this solution is also helpful in removing deposits or particulate matter.
2. Clean the torch periodically by soaking in 10% HNO₃.
3. Sample pump tubing should be inspected every day and changed when showing signs of wear.

4. The purge window may fog slightly over time. Remove and clean with methanol.
5. Check the water level in the chiller.
6. A Perkin Elmer service engineer performs scheduled periodic maintenance every six months.
7. All other maintenance shall be documented in the instrument maintenance log. If a particular problem occurs, document the problem and the solution.

CALCULATIONS

1. Sample Concentration -

$$\text{mg/L or mg/kg analyte in sample} = A \frac{V_f}{V_i} \times d$$

A = mg/L analyte in processed sample
 Vf = final volume of sample (ml)
 Vi = initial volume (ml) or weight (g) of sample
 d = dilution factor (if required)
 The units may be converted to ug/L or ug/kg

2. Matrix spike and Matrix Spike Duplicate Recovery

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

SSR = spiked sample result
 SR = sample result
 SA = spike added

3. Matrix Duplicate Relative Percent Difference (RPD)

$$\text{RPD} = \frac{SR - SDR}{\frac{(SR+SDR)}{2}} \times 100$$

SR = Sample result
 SDR = sample duplicate result
 $\frac{(SR+SDR)}{2}$ = average of SR and SDR

TABLE I

ELEME NT	WAVELENGTH	WAVELENGTH	CALIBRATION STANDARD CONC mg/L
	5300DV	4300DV*	
Ag	328.067	328.072	1.00
Al	308.218	308.219	10.0

ELEMENT	WAVELENGTH	WAVELENGTH	CALIBRATION STANDARD CONC mg/L
	5300DV	4300DV*	
As	193.696	193.694	1.00
B	249.672	249.677	5.00
Ba	233.527	233.524	1.00
Be	313.105	313.104	1.00
Ca	315.887	315.887	10.0
Cd	214.438	214.436	1.00
Ce	418.660	418.660	1.00
Co	228.613	228.617	1.00
Cr	267.706	267.713	1.00
Cu	324.751	324.753	1.00
Fe	259.938	259.938	10.0
K	766.496	766.526	20.0
Li	670.783	670.813	1.00
Mg	279.078	279.076	10.0
Mn	257.605	257.609	1.00
Mo	202.031	202.032	1.00
Na	589.597	589.622	40.0
Ni	231.605	231.602	1.00
Pb	220.352	220.353	1.00
Sb	206.830	206.834	1.00
Se	196.024	196.026	1.00
Si	251.609	251.612	1.00
Sn	189.928	189.924	1.00
Sr	421.560	421.563	1.00
Ti	337.277	337.277	1.00
Tl	190.800	190.799	1.00

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ELEM NT	WAVELENGTH	WAVELENGTH	CALIBRATION STANDARD CONC mg/L
	5300DV	4300DV*	
V	290.878	290.881	1.00
Zn	213.857	213.856	1.00
Zr	343.821	343.818	1.00

*Al, Ca, Fe, Na, Li, K, Sr, Mg are analyzed in the radial mode

METHOD
PERFORMANCE
(METHOD 6010)

- 1) In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 of Method 6010 lists the true values, the mean reported values, and the mean percent relative standard deviations.
- 2) Performance data for aqueous solutions and solid samples from a multi-laboratory study (9) are provided in Tables 5 and 6 of Method 6010.

METHOD
PERFORMANCE
(METHOD 200.7)

- 1) Listed in Table 4 of Method 200.7 are typical single laboratory total recoverable MDLs determined for the recommended wavelengths using simultaneous ICP-AES and the operating conditions given in Table 5 of Method 200.7. The MDLs were determined in reagent blank matrix (best case situation). PTFE beakers were used to avoid boron and silica contamination from glassware with the final dilution to 50 ml completed in polypropylene centrifuged tubes. The listed MDLs for solids are estimates and were calculated from the aqueous MDL determinations.
- 2) Data obtained from single laboratory method testing are summarized in Table 6 of Method 200.7 for five types of water samples consisting of drinking water, surface water, ground water, and two wastewater effluents. The data presented cover all analytes except cerium and titanium. Samples were prepared using the procedure described in Section 11.2. For each matrix, five replicate aliquots were prepared, analyzed and the average of the five determinations used to define the sample background concentration of each analyte. In addition, two pairs of duplicates were fortified at different concentration levels. For each method analyte, the sample background concentration, mean percent recovery, standard

deviation of the percent recovery, and relative percent difference between the duplicate fortified samples are listed in Table 6 of Method 200.7. The variance of the five replicate sample background determinations is included in the calculated standard deviation of the percent recovery when the analyte concentration in the sample was greater than the MDL. The tap and well waters were processed in Teflon and quartz beakers and diluted in polypropylene centrifuged tubes. The nonuse of borosilicate glassware is reflected in the precision and recovery data for born and silica in those two sample types.

- 3) Data obtained from single laboratory method testing are summarized in Table 7 of Method 200.7 for three solid samples consisting of EPA 884 Hazardous Soil, SRM 1645 River Sediment, and EPA 286 Electroplating Sludge. Samples were prepared using the procedure described in Section 11.3. For each method analyte, the sample background concentration, mean percent recovery of the fortified additions, the standard deviation of the percent recovery, and relative percent difference between duplicate additions were determined as described in Section 13.2. Data presented are for all analytes except cerium, silica and titanium. Limited comparative data to other methods and SRM materials are presented in reference 23 of Section 16.0.
- 4) Performance data for aqueous solutions independent of sample preparation from a multi-laboratory study are provided in Table 8 of Method 200.7.
- 5) Listed in Table 9 of Method 200.7 are regression equations for precision and bias for 25 analytes abstracted from EPA Method Study 27, a multi-laboratory validation study of Method 200.7. The equations were developed from data received from 12 laboratories using the total recoverable sample preparation procedure on reagent water, drinking water, surface water and 3 industrial effluents. For a complete review and description of the study see reference 16 of Section 16.0.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
METALS
STANDARD OPERATING PROCEDURE

PROCEDURE: MET-005
PAGE: 1 OF 4
EFFECTIVE DATE: 09/18/07
APPROVED BY: *MAT*
QA/QC APPROVED: *AM*

SUBJECT

SCOPE AND APPLICATION

This digestion procedure is designed for the preparation of aqueous samples, TCLP extracts and EPTOX extracts. This is a vigorous digestion with nitric and hydrochloric acid for the determination of total metals by inductively coupled plasma spectroscopy (ICP).

MATRIX

Water

SAFETY

1. All employees will view a video at a safety orientation prior to performing work within the laboratory.
2. Whenever any acids are used, safety glasses must be worn. Acid resistant gloves shall be worn at all times.
3. Evaporation and/or digestion with acids must be performed under a well-ventilated acid resistant fume hood.
4. Each chemical compound must be treated as a potential hazard. Exposure to these chemicals must be reduced to the lowest possible level by means of fume hoods, respirators and protective clothing.
5. A reference file of material safety data sheets (MSDS) are available to all personnel to provide knowledge on safe handling of chemicals used in this procedure.

REFERENCES

SW846 3010A

PRESERVATION

Dissolved Metals -- Filtered through a 0.45 μ m membrane filter then HNO₃ to pH <2
Total Metals -- HNO₃ to pH <2

HOLDING TIMES

Dissolved Metals - 6 months
Total Metals - 6 months

DEFINITIONS

See SOP GEN-016

APPARATUS

250 ml beakers
Ribbed and plain watchglasses
50 ml graduated cylinders
Specimen containers

Hot Plate
CPI Mod Block - Digestion Block
70 mL Digestion Tubes
Mechanical pipet and clear plastic pipet tips
Wide range pH paper

REAGENTS
AND STANDARDS

Label all containers and squeeze bottles with reagent ID, lot, and expiration date.
All standards used are pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.
Nitric Acid -- Trace Metal grade
HCl -- Trace Metal grade
Spiking Solution for samples and LCS
 GCAL-1: certified custom solution
 GCAL-2: certified custom solution
Deionized water

PROCEDURE

BLOCK DIGESTION SYSTEM

1. Record all sample information in the metals preparation form (attached).
2. Measure 50 ml of a homogenized sample in a 70 mL digestion tube labeled with sample number, date, and prep for all TCLP liquid and aqueous samples. For TCLP organic liquids measure 5mL of sample. Add 1.5 ml concentrated HNO₃ and cover with ribbed watch glass or equivalent.
3. Place tubes in the digestion block. Select Block A or B. Set temperature to the required setting for a temperature of 90-95°C. "B" key for up and "E" key for down. When the desired temperature is reached; Press Enter (C key): 2 hours will show for timer; Press Enter (C key): start time will show; Press Enter (C key): Block will heat to 95°C and maintain heat for 2 hours. Place a thermometer in an uncovered digestion tube to monitor temperature. Record the temperature in the MOD Block Temperature Logbook.
4. Evaporate to about 5 mL without boiling. (Heat may be increased to get a gentle reflux.)

5. Allow sample to cool. Add 1.5 mL of concentrated HNO_3 .
6. Reflux and add additional HNO_3 until the reaction is complete.
7. Evaporate to small volume, cool and add 5 mL of 1:1 HCl (1mL 1:1 HCl per 10mL of final solution).
8. Reflux 15 minutes.
9. Cool and adjust final volume to 50 mL with DI H_2O .
10. The sample is now ready for analysis. Deliver to the metals laboratory with a copy of the prep log.

QUALITY CONTROL With each batch twenty or fewer samples, the following will be digested:

- a. Method Blank (BLK) -- DI + reagents
- b. Laboratory Control Sample (LCS) -- DI + reagents -- add 0.25 ml of GCAL-1 and 0.25 ml of GCAL-2. (Check to be sure all elements requested to be run are contained in the spiking solutions. If not, add them separately from stock solutions.)
- c. Method Blank (extracted blank for TCLP) - TCLP extracts only
- d. Duplicate sample
- e. Matrix Spike -- add 0.25 ml of GCAL-1 and 0.25 mL of GCAL-2. (Check to be sure all requested elements are contained in the spiking solutions. If not, add them separately from stock solutions.)
- f. Matrix Spike Duplicate (performed only if required for a project or requested by the client) -- add 0.25 ml of GCAL-1 and 0.25 mL of GCAL-2. (Check to be sure all requested elements are contained in the spike solutions. If not, add them separately from stock solutions.)
- g. Record the laboratory ID for all standards and reagents

in the logbook.

- h. Specific projects may require additional or specific QC, and must be followed for applicable samples. See Sop GEN-019.

METHOD
PERFORMANCE

METHOD
PERFORMANCE

3010A
No Data Provided

POLLUTION
PREVENTION

See QAPP Section 10.2

WASTE
MANAGEMENT

See SOP GEN-009

ICP SAMPLE PREPARATION FORM

EXTRACTION DATE/TIME:					BATCH NO:		
MATRIX:		WATER <input type="checkbox"/>	SOIL <input type="checkbox"/>	TCLP EXT <input type="checkbox"/>	ORGANIC <input type="checkbox"/>	METHOD:	200.7 <input type="checkbox"/> 3010A <input type="checkbox"/> 3050B <input type="checkbox"/> 3051 <input type="checkbox"/>
CLIENT	CLIENT ID	GCAL ID	INITIAL VOL/WT mL g	FINAL VOLUME (mL)	COMMENTS	REAGENTS/ STANDARDS	
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
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22							
23							
24							
25							
26							
27							
28							

COMMENTS:

BLOCK ID	TECHNICIAN	DATE
REPIPET BOTTLES VERIFIED	REVIEW	DATE

STANDARD OPERATING PROCEDURE
REVIEW FORM

GCAL
Standard Operating Procedure for

Procedure: MEF-005
Revision:
Effective Date: 9-18-07

Reviewed By/Date: JEL 8-23-08

Approved By/Date: JDT 8/25/08

No changes needed.

GULF COAST ANALYTICAL LABORATORIES, INC.
SAMPLE ADMINISTRATION
STANDARD OPERATING PROCEDURE

PROCEDURES: SAD-001
PAGE: 1 OF 2
EFFECTIVE DATE: 04/03/09
APPROVED BY: JDT
QA/QC APPROVED: MAP

SUBJECT SCOPE AND APPLICATION

Log-in is responsible for receiving all samples submitted for analyses. Maintaining chain of custody records for all samples, logging appropriate samples into the LIMS and defining analyses and due dates are all part of the log-in daily routine.

DEFINITIONS See SOP GEN-016

PROCEDURES

1. Receive all samples from clients and/or couriers, maintain proper chain of custody records, and confirm sample identifications and number of samples submitted. Compare information on the chain of custody to the labels on the containers. Check for leakage, cracked or broken closures or containers, and other apparent sample integrity problems. Document any of the above problems on a Login Discrepancy Form and forward to the Project Manager. Take temperature of all ice chests with the Infrared thermometer. Select a representative sample container from the ice chest and read temperature by holding the IR gun flat against the label on the side of the container. For multiple cooler shipments, a representative sample will be chosen from each ice chest to check the temperature. Apply any applicable correction factor to the temperature. Samples received outside the acceptance range of $\leq 6^{\circ}\text{C}$ should be documented on the chain of custody and the sample preservation information sheet. For multiple cooler shipment temperature excursions, all sample containers from the particular ice chest containing the excursion will be cataloged and reported to the project manager. Samples received within a relatively short time after collection may not have reached the required temperature although they are thermally preserved. Record a comment on the Preservative Checklist/Cooler Receipt (PC/CR) form if this situation applies. Make notes of any concerns or abnormalities on the chain of custody and sample preservation information sheet. NOTE: The sample temperature should be taken at the label so that emissivity correction of different surfaces does not have to be accounted for. This will help to ensure that all GCAL login personnel consistently and accurately check the temperature. Also, be advised that many factors including the distance-to-spot ration and field of view may effect the accuracy of the measurement. Please see the manufacturer's instruction for proper operation of the IR gun.
2. Enter samples into the LIMS according to proper established LIMS procedures. Due date, analyses, sample date, sample time, comments, concerns, and/or cautions must be entered.
3. Sort and label all containers.
4. Verify the pH of all pH preserved samples (excluding VOA'S and Oil & Grease). If a vial for preservation checks for VOA's is submitted it is also checked at log-in. For all CH2M Hill ARCEE VOA vials the pH is checked at log-in at the time of

of sample. Place a drop on pH paper. If pH is not <2 or is >12, pH is adjusted using the short-range pH paper. Do not adjust VOA samples for pH. If the pH is not <2, then the VOA sample should be logged in for a 7-day hold time. If an aliquot of the sample is not preserved, sample preservation is marked unacceptable on the Preservative Checklist/Cooler Receipt (PC/CR). The client will be contacted if required, regarding instructions on how to proceed. If samples are preserved at the laboratory, follow the requirements for appropriate preservatives and volumes listed on the sample kit request form. Document this preservation in the comment section on the PC/CR. The laboratory ID of the preservative will also be recorded. **NOTE: Handle all samples with caution. Ventilation hoods should be used when containers are opened.**

5. For all metals samples that are preserved at sample receipt, hold the samples in the holding cooler for at least 24 hours before releasing to the laboratory for analysis.
6. Visually inspect VOA vials for bubbles. The presence of any bubbles should be documented in the comment section for the aliquot on the PC/CR. The aliquot is identified as unacceptable if the bubbles are greater than 1% of the vial volume (usually a bubble size of 4 mm in diameter).
7. Page analysts for all immediate parameters as determined by holding time and/or priorities previously defined by client services.
8. Original chain of custodies, preservation sheets, and log-in summaries are submitted to the Project Manager for review.
9. Make corrections/changes as indicated on the Login Discrepancy form. All changes must be documented in writing.
10. Assist sample custodian in any shipment and/or bottle kit preparation.
11. Generate courier schedule each day for pick-ups or deliveries. Provide couriers and project managers with a copy of daily schedule.
12. Direct couriers throughout the day with additional sample pick-ups that are received from project managers.

POLLUTION PREVENTION See QAPP Section 13.2

WASTE MANAGEMENT See SOP GEN-009

Uncontrolled Copy - For Reference Only

PRESERVATION CHECKLIST / COOLER RECEIPT

Gulf Coast Analytical Laboratories, Inc.

WO:
 Desc:
 Work ID:
 Project Seq:
 Client:
 Profile:

Type:
 Report:
 Status:
 Created:
 QA:
 PO:

WORKORDER SAMPLES

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
No containers for this Lab ID			<input type="checkbox"/>						
Container ID	Type	Preservative	A	U	N/A	A	U	N/A	CONTAINER CONDITION
No containers for this Lab ID			<input type="checkbox"/>						
Container ID	Type	Preservative	A	U	N/A	A	U	N/A	CONTAINER CONDITION
No containers for this Lab ID			<input type="checkbox"/>						
Container ID	Type	Preservative	A	U	N/A	A	U	N/A	CONTAINER CONDITION
No containers for this Lab ID			<input type="checkbox"/>						

A = ACCEPTABLE
 U = UNACCEPTABLE
 N/A = NOT APPLICABLE

COOLER (S) TEMPERATURE A U LIMIT = 4C + 1 - 2C
 MAXIMUM VOLATILE HEADSPACE BUBBLE 6MM

Custody Seal		
used	<input type="checkbox"/> Yes	<input type="checkbox"/> No
in tact	<input type="checkbox"/> Yes	<input type="checkbox"/> No

LABEL(S) VERIFIED _____ CUSTODIAN _____

GULF COAST ANALYTICAL LABS
METALS
STANDARD OPERATING PROCEDURE

PROCEDURE: MET-008
PAGE: 1 OF 8
EFFECTIVE DATE: 8/25/2008
APPROVED BY: MAP
QA/QC APPROVED: JDT

SUBJECT SCOPE AND APPLICATION

This procedure is used for the determination of dissolved or total mercury in aqueous samples, TCLP extracts, EPTOX extracts, Industrial Hygiene samples, soils, solids, and domestic and industrial wastes by the cold vapor atomic absorption technique. A digestion procedure is required for all samples and standards.

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 254 nm. Absorbance by the mercury cold vapor is measured using a photocell detector with a wide dynamic range.

MATRIX Water and Solids

REFERENCES EPA Method 245.1/245.2
SW846 Methods 7470A and 7471B
NIOSH Method 6009
Perkin Elmer FIMS 400 Instrument Manual

PRESERVATION & HOLDING TIME Aqueous Samples:
Dissolved Hg - Filtered, HNO₃ to pH <2, 28 days
Total Hg - HNO₃ to pH <2, 28 days

TCLP/EPTOX Hg - 4°C, 28 days from field collection to extraction, 28 days from digestion to analysis

Solid Samples - 4°C, 28 days

DEFINITIONS See SOP GEN-016

SAFETY 1. All employees will view a video at a safety orientation prior to performing work within the laboratory.
2. Whenever any acids are used, safety glasses must be worn. When working with acids, a face shield and an

acid resistant apron should be worn. Acid resistant gloves should be worn at all times.

3. Evaporation and/or digestion with acids must be performed under a well-ventilated acid resistant fume hood.
4. Each chemical compound must be treated as a potential hazard. Exposure to these chemicals must be reduced to the lowest possible level by means of fume hoods, respirators and protective clothing.
5. A reference file of material safety data sheets (MSDS) are available to all personnel to provide knowledge on safe handling of chemicals used in this procedure.

APPARATUS

Perkin Elmer FIMS 400 Mercury Analyzer with computer and printer

Pump tubing: sample, drain assembly, reductant, carrier

Autosampler standard cups and sample tubes

100 ml volumetric flasks

INTERFERENCES

1. Sulfide -- Interferences from concentrations as high as 20 mg/L sulfide are eliminated by adding potassium permanganate.
2. Copper can interfere at concentrations above 10 mg/L.
3. High chloride concentrations in seawaters, brines, and industrial effluents require additional permanganate. During the oxidation step, free chlorine is formed and absorbs radiation at 253.7 nm.
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.

REAGENTS AND
STANDARDS

1. 10% HCl for rinse bath
2. Stannous chloride -- 20 g stannous chloride in 5% HCl (trace metal grade) diluted to 2 liter for a 1% solution. Mix well to dissolve the stannous chloride.

3. Sodium chloride - hydroxylamine sulfate solution:
Dissolve 120g of sodium chloride and 120g of hydroxylamine sulfate in deionized water and dilute to 1 liter.
4. 3% HCl solution for carrier
5. 1000 mg/L stock standard (commercially obtained)
6. 1 mg/L mercury working standard - Add 100 ul of the stock standard to a 100 ul volumetric flask. Dilute to volume with 2% HNO₃. Record in the standard preparation logbook. This standard should be prepared every two weeks.
7. Second source stock standard (commercially obtained) is used to prepare a 1 mg/L standard for independent verification of the calibration standard (ICV).

PROCEDURE

1. Open the FIMS 400 software by double clicking on the AAWINLAB icon. Turn Hg lamp on. Allow the lamp to warm up for at least 30 minutes.
2. Check pump tubing on the motor. Change sample tubing daily. Change reductant, carrier, and waste tubing as needed.
3. Wash and acid rinse the rinse bath and fill with 10% HCl as needed.
4. Place reductant tube into the container of stannous chloride.
5. Place the carrier tube into the container of 3% HCl.
6. Change the filter on the liquid gas separator as needed.
7. Adjust the argon flow to 80-100 psi.
8. Prepare a bench sheet -- record the laboratory ID for the each calibration standard, the ICV, the stannous chloride and the sodium chloride -- hydroxylamine solution. The bench sheet used is attached.

9. Prepare digested standards and samples for analysis by adding 4 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate (Decolorize).
10. Add standards to the standards rack. See autosampler layout diagram for positions. Standards and their positions are: 0.0 ug/L (blank)-1; 0.2 ug/L-2; 0.5 ug/L-3; 2.0 ug/L-4; 5.0 ug/L-5; 10.0 ug/L-6.
11. Fill sample tubes with the following quality control samples:

<u>Position</u>	<u>Standard</u>
9	5 ug/L - Independent Source (ICV)
1	0.0 (ICB)
5	CCV
1	CCB

12. Fill sample tubes with samples (3 - 2 inch from top) and place in the position in the rack corresponding to their positions on the bench sheet.
13. To type in sample descriptions for analysis, bring up the sample information file. Type in the sample ID next to the corresponding autosampler location. Autosampler positions available for the Perkin Elmer AS-90 are 17 to 106. Save the sample information file, and then close this window.
14. Open the automated analysis window and select the mercury method to be used. Type in the autosampler locations to be analyzed, and select the result data set to be used.
15. Select the analyze tab at the bottom of the automated analysis window. Click on analyze all to calibrate, and then run samples.

SHUT DOWN
PROCEDURE

1. Let instrument run for a few minutes after the last sample is completed.

2. Turn off the lamp.
3. Refill the rinse bath.
4. Dispose of the standard and sample remaining in the tubes. Pour in the metals waste container.

QUALITY CONTROL

1. The correlation coefficient should be ≥ 0.9995 . If the calibration results are unacceptable, rerun the standards to verify results. Prepare new calibration standards if necessary. When acceptable results are achieved, accept the calibration.
2. The ICV recovery must be 90-110%.
3. A CCV and CCB will be analyzed every 10 samples. The CCV recovery must be 80-120%. If a CCV is outside control limits, the previous ten samples must be reanalyzed. The CCB must be $<$ the reporting limit and $>2X$ the - reporting limit. The results may be reported with a narrative if the associated samples do not have a hit.
4. A Laboratory Control Sample (LCS) is prepped with each batch of 20 samples. A Laboratory Control Sample Duplicate (LCSD) is prepped if required for the project or if insufficient sample is available for a duplicate or MS/MSD. The recovery must be 80-120%. The LCS/LCSD RPD should be $<20\%$. If the recovery is outside the control limits, all samples in the batch must be re-digested and re-analyzed.
5. A matrix duplicate is prepped with every 20 samples. A The RPD for duplicates should be $<20\%$ unless the sample concentrations are less than five times the detection limit or the sample matrix is nonhomogeneous.
6. Matrix spike (MS) and matrix spike duplicate (MSD-if required for project) recoveries should be 75-125%. The MS/MSD RPD should be $<20\%$. If the recovery is outside these limits, the data should be flagged. A spike is not applicable if the sample concentration is greater than 4 times the spike level. If a TCLP extract spike recovery for a regulated metal is $<50\%$ and the sample concentration is within 20% of the regulatory

level the sample must be reanalyzed by the method of standard additions. Dilutions may be performed to eliminate an interference that may cause unacceptable spike recoveries. If recoveries are improved by the dilution, the original sample must also be diluted. It may also be necessary to re-digest a diluted aliquot of the sample.

7. If the apparent concentration of a sample is greater than the highest calibration standard, the sample should be diluted and rerun.
8. Dilution Test - Perform a 5x dilution on one sample per batch, generally on the sample used for the MS. For the test to be applicable, the concentration should be greater than 50 times the MDL. The neat sample concentration and five times the diluted sample concentration should agree within 10%. If not, a chemical or physical interference should be suspected.
9. Additional project specific criteria may apply to projects and may be more stringent. See SOP GEN-019, and check with project manager for additional guidance.

MAINTENANCE

1. Change reductant and drain tubing as needed.
2. Replace mercury lamp as needed. (Average life -- 4 months to 1 year) A transmittance value of <400,000 for the reference intensity and sample intensity in systems diagnostics indicates a weak lamp.
3. Clean the optical cell and lenses as needed. Relative absorbances of standards may differ significantly from previous calibrations when the cell is dirty.
4. Replace the liquid gas separator every 1-3 years.
5. Record all maintenance in the instrument maintenance logbook.
5. Call Perkin Elmer Service Department for specific troubleshooting guidelines that may require specialized maintenance.

CALCULATIONS

1. For Waters or TCLP extracts --
Sample Concentration in ug/L = instrument reading x d
Sample Concentration in mg/L = A x d

A = ug/L analyte in processed sample/1000
d = dilution factor (if required)

The raw data units are in ug/L - divide by 1000 to convert to mg/L.

2. For Solids
Sample Concentration in mg/kg = $30 \times \frac{A \times d}{W}$

A = mg/L analyte in processed sample
W = initial weight of sample (g)
d = dilution factor (if required)
30 = final volume (prep)

The raw data units are in ug/L - divide by 1000 to convert to mg/L.

3. Sample concentration -- Industrial Hygiene Samples

Total ug analyte in sample = A x 50 ml x d

A = ug/ml (mg/L) analyte in processed sample
50 ml = final volume of sample
d = dilution factor (if required)

4. Matrix spike recovery

% Recovery = $\frac{(SSR - SR) \times 100}{SA}$

5. Matrix Duplicate Relative Percent Difference (RPD)

RPD = $\frac{|SR - SDR| \times 100}{\frac{(SR+SDR)}{2}}$

SR = Sample result
SDR = sample duplicate result
(SR+SDR) = average of SR and SDR

REPORTING
LIMITS

Waters: 0.0002 mg/L

Solids: 0.01 mg/kg

METHOD

PERFORMANCE

- 1) In a single laboratory (SEWL), using distilled water standards at concentrations of 0.5, 1.0, 2.0, 5.0, 10.0 and 20.0 ug Hg/L, the standard deviations were ± 0.04 , ± 0.07 , ± 0.09 , ± 0.020 , ± 0.40 and ± 0.84 ug/L, respectively.
- 2) In a single laboratory (SEWL), using surface water samples spiked with ten organic mercurials at the 10 ug/L level, recoveries ranged from 87 to 117%. Recoveries of the same ten organic mercurials in distilled water at the 10 ug/L level, ranged from 92% to 125%.
- 3) The following standard deviations on replicate sediment samples were recorded at the indicated levels; 0.29 ug/G ± 0.02 and 0.82 ug/G ± 0.03 . Recovery of mercury at these levels, added as methyl mercuric chloride, was 97% and 94%, respectively.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABS
METALS
STANDARD OPERATING PROCEDURE

PROCEDURE: MET-006
PAGE: 1 OF 6
EFFECTIVE DATE: 10/13/2008
APPROVED BY: *MAP*
QA/QC APPROVED: *JDT*

SUBJECT SCOPE AND APPLICATION

These digestion procedures are used for the preparation of aqueous samples, TCLP extracts, EPTOX extracts, soils, solids, and domestic and industrial wastes for mercury analysis.

MATRIX Water and Solid

REFERENCES EPA-245.1/245.2
SW846-7470A/7471B

PRESERVATION &
HOLDING TIME

Aqueous Samples:
Dissolved Hg - Filtered, HNO₃ to pH <2, 28 days
Total Hg - HNO₃ to pH <2, 28 days

TCLP/EPTOX Hg - 4°C, 28 days from field collection to extraction, 28 days from digestion to analysis

Solid Samples - 4°C, 28days

DEFINITIONS See SOP GEN-016

SAFETY

1. All employees will view safety video orientation prior to performing work within the laboratory.
2. Whenever any acids are used, safety glasses must be worn. When working with acids, a face shield and an acid resistant apron should be worn. Acid resistant gloves should be worn at all times.
3. Evaporation and/or digestion with acids must be performed under a well-ventilated acid resistant fume hood.
4. Each chemical compound must be treated as a potential hazard. Exposure to these chemicals must be reduced to the lowest possible level by means of fume hoods, respirators and protective clothing.

5. A reference file of material safety data sheets (MSDS) are available to all personnel to provide knowledge on safe handling of chemicals used in this procedure.

REAGENTS,
STANDARDS, AND
APPARATUS

1. 70 mL Digestion tubes
2. Deionized water - monitored daily
3. Concentrated Sulfuric Acid (H_2SO_4) - trace metals grade
4. Concentrated Nitric Acid (HNO_3) - trace metals grade
5. Concentrated Hydrochloric Acid (HCl)
6. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated Hydrochloric acid to one volume of Nitric acid.
7. 5% Potassium Permanganate - dissolve 5g of Potassium Permanganate in 100ml of DI water
8. 5% Potassium Persulfate - dissolve 5g of Potassium Persulfate in 100ml of DI water
9. Mechanical pipette - clear, plastic pipette tips, accuracy of 2%.
10. 50 mL graduated cylinder
11. CPI Mod Block - Digestion block
12. 1 mg/L Hg working standard - prepared by the metals laboratory monthly
13. 0.1 mg/L Mercury working standard - prepared by the metals laboratory daily.
14. Electronic balance capable of weighing to 0.01g
15. Teflon boiling beads (or equivalent)

PROCEDURE

Aqueous, TCLP extracts, EPTOX extracts (except organic samples)

1. Prepare a page in the metals preparation sheet. Record sample ID's.
2. Label each digestion tube with sample ID, date, and matrix.
3. Shake sample well and measure 20 ml of homogenized sample in the digestion tube. Prepare all QC samples as listed in the Quality Control Section.
4. Add 1.0 ml of H_2SO_4 and 0.5 mL concentrated HNO_3 , mixing after each addition.
5. 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.
6. Add 2 ml Potassium Persulfate solution and heat at 95°C

for 2 hours.

7. Perform steps #1-#6 for 20 mL aliquots of a blank (deionized water), 10 $\mu\text{g/L}$, 5 $\mu\text{g/L}$, 2 $\mu\text{g/L}$, 0.5 $\mu\text{g/L}$, and 0.2 $\mu\text{g/L}$ calibration standards and a 5 $\mu\text{g/L}$ independent calibration verification standard. Standards are prepared using a 0.1 ppm stock solution prepared daily in the metals lab.

The mercury calibration curve is prepared as follows:

0.2 $\mu\text{g/L}$: 40 μL	0.1 ppm calibration standard
0.5 $\mu\text{g/L}$: 100 μL	0.1 ppm calibration standard
2.0 $\mu\text{g/L}$: 400 μL	0.1 ppm calibration standard
5.0 $\mu\text{g/L}$: 1000 μL	0.1 ppm calibration standard
10.0 $\mu\text{g/L}$: 2000 μL	0.1 ppm calibration standard
5.0 $\mu\text{g/L}$: 1000 μL	0.1 ppm independent standard

Dilute these solutions to 20 ml with deionized water. Multiple aliquots of each standard may be prepared.

8. Release sample to the metals laboratory. Sodium Chloride-hydroxylamine sulfate will be added in the analytical laboratory.

Solids, Soils, Domestic and Industrial Wastes (except organic samples)

1. Prepare a page in the metals preparation sheet. Record sample ID's.
2. Label each digestion tube with sample ID, date and matrix.
3. Homogenize sample by pouring the contents of the sample onto a piece of butcher paper, chop and mix with a tongue depressor or spatula. Remove any foreign object such as sticks, leaves, or rocks. Weigh 0.50g - 0.60g of the homogenized sample and place in the digestion tube. A minimum weight may be required to meet the project required detection limit. Check with the metals supervisor or project manager to determine if a minimum weight is required. Record the weight to the nearest 0.01 g in the prep sheet. Prepare QC samples as listed in the quality control section.
4. Add 5 ml of DI water and add 5 mL of Aqua Regia.
5. Heat the sample at 95°C for 2 minutes.
6. Cool and add 30 mL DI water and 15 mL 5% Potassium

Permanganate solution, mix thoroughly. Add additional Potassium Permanganate as needed to maintain purple color. Record additional volume added on prep sheet.

7. Digest the sample for 30 minutes at 95°C.
8. Perform steps 4-7 for 30 mL aliquots of blank (deionized water), 10 µg/L, 5 µg/L, 2 µg/L, 0.5 µg/L, and 0.2 µg/L calibration standards and a 5µg/L independent calibration verification standard. Standards are prepared using a 0.1 ppm solution prepared daily in the metals laboratory.

The mercury calculation curve is prepared as follows:

0.2 µg/L	:	60 µL	0.1 ppm calibration standard
0.5 µg/L	:	150 µL	0.1 ppm calibration standard
2.0 µg/L	:	600 µL	0.1 ppm calibration standard
5.0 µg/L	:	1500 µL	0.1 ppm calibration standard
10.0 µg/L	:	3000 µL	0.1 ppm calibration standard
5.0 µg/L	:	1500 µL	0.1 ppm independent standard

Dilute these solutions to 30 mL with deionized water. Multiple aliquots of each standard may be prepared.

9. Release samples to the metals laboratory. Sodium Chloride-hydroxylamine sulfate will be added in the analytical laboratory.

Organic samples including TCLP Liquid extracts and UTS Liquid extracts: The procedure has been modified to more thoroughly digest samples of this matrix type.

1. Prepare a page in the metals preparation sheet. Record sample ID's.
2. Label each digestion tube with sample ID, date, and matrix.
3. Weigh 0.6g (Total Hg), 0.6 mL (TCLP or UTS Hg) of well-mixed sample and place in digestion tube. Record sample weight to the nearest 0.01g in prep sheet. Prepare QC samples as listed in the Quality Control Section.
4. Add 5 mL of concentrated HNO₃ and 5 mL of concentrated HCl.
5. Heat in the Mod Block until the fumes are clear or unchanging. Additional acid may be added to complete the digestion.
6. Add 30 mL of deionized water.
7. Add 15 mL Potassium Permanganate solution and mix

- thoroughly.
8. Digest 30 minutes at 95°C.
 9. Remove from the digestion block and allow to cool.
 10. These will be analyzed using calibration standards prepared by the solid digestion procedure.
 11. Release sample to the metals laboratory. Sodium Chloride-hydroxylamine sulfate will be added in the analytical laboratory.

QUALITY CONTROL

1. Batches will be defined by sample matrix:
 - Aqueous
 - TCLP extracts
 - EPTOX extracts
 - Soils/Sludges
 - Oily wastes
 - Client specific batch
2. Prepare a reagent blank with each batch of samples (20 or fewer samples). A method blank using filtered deionized water (liquid TCLP) or the appropriate extraction fluid is used for extracts. Reagent blanks for solids prepare by weighing approximately 0.6 g of Teflon boiling beads. Document exact weight.
3. Prepare a matrix duplicate for every 20 samples.
4. Prepare a matrix spike (MS) for every 20 samples. A matrix spike duplicate (MSD) will be prepared if required for the project.

Aqueous/extracts - add 100 μ L of the 1 mg/L Hg standard (5 μ g/L)

Non-Aqueous - add 150 μ L of the 1 mg/L Hg standard (5 μ g/L in solution)
5. Prepare a laboratory control standard (LCS) with every 20 samples. A laboratory control standard duplicate (LCS D) will be prepared if required for the project or if insufficient sample is available for a matrix duplicate or MS/MSD.

Aqueous/extracts

Add 100 μ L of the 1 mg/L working standard (5 μ g/L)

A LCS prepared with filtered deionized water or the appropriate extraction fluid is used for extracts.
Non-aqueous

Weigh approximately 0.6 g of Teflon boiling beads, document exact weight on prep sheet. Add 150 μ L of the 1 mg/L working standard (5 μ g/L in solution)

6. Record the laboratory ID of the standard and reagents in the prep sheet.

METHOD

PERFORMANCE

- 1) In a single laboratory (SEWL), using distilled water standards at concentrations of 0.5, 1.0, 2.0, 5.0, 10.0 and 20.0 μ g Hg/L, the standard deviations were ± 0.04 , ± 0.07 , ± 0.09 , ± 0.020 , ± 0.40 and ± 0.84 μ g/L, respectively.
- 2) In a single laboratory (SEWL), using surface water samples spiked with ten organic mercurials at the 10 μ g/L level, recoveries ranged from 87 to 117%. Recoveries of the same ten organic mercurials in distilled water at the 10 μ g/L level, ranged from 92% to 125%.
- 3) The following standard deviations on replicate sediment samples were recorded at the indicated levels; 0.29 μ g/G ± 0.02 and 0.82 μ g/G ± 0.03 . Recovery of mercury at these levels, added as methyl mercuric chloride, was 97% and 94%, respectively.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABS
GC - PESTICIDES METHOD 8081
STANDARD OPERATING PROCEDURE

PROCEDURES: GC-013
PAGES: 1 OF 13
EFFECTIVE DATE: 10/07/2008
APPROVED BY: *MAP*
QA/QC APPROVED: *JOT*

SUBJECT

SCOPE AND APPLICATION

This method provides gas chromatographic procedures for the detection of ppb levels of certain Organochlorine pesticides. Prior to the use of this method, appropriate extraction techniques must be used. The extraction is described in EXT-002 for solid samples and EXT-010 for aqueous samples. An aliquot of the sample extract is injected into a GC equipped with an ECD detector.

MATRIX

Water and Solid

REFERENCE

SW-846 Method 8081B, 8000C
For North Carolina and South Carolina projects SW-846 8000B must be used instead of SW-846 8000C.

PRESERVATIVE

Cool 4°C, protect from light

HOLDING TIME

Water - From collection to extraction - 7 days
From extraction to analysis - 40 days
Solid - From collection to extraction - 14 days
From extraction to analysis - 40 days

DEFINITIONS

See SOP GEN-016

SAFETY

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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

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.

INTERFERENCES

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.

INSTRUMENTATION

Gas Chromatograph should be suitable for split-less or on-column injection. The system should be equipped with an electron capture detector. The instruments used are

Agilent Technology 6890N GC/ECD.

The data system must be capable of time stamping, all data produced, with the correct date and time. The data system employed is Target.

COLUMNS

Recommended Columns are
XLB or similar phase 30m X 0.32mm ID
35MS or similar phase 30m X 0.32mm ID

REAGENTS

Hexane-pesticide grade. Store away from sources of Phthalates.

STANDARDS

1. Purchasing Standards: All standards shall be purchased from a certified vendor. The ICV shall be from a secondary source.
2. The 8081 stock standard is a certified standard containing all single component analytes at varying concentrations for calibration. Add 625uL and dilute to 50mL with Hexane to make the working standard.
3. The Toxaphene stock standard is purchased certified to contain 5000ug/mL. A working standard of 5000ug/L is prepared by spiking 50uL of the stock standard and diluting to 50mL in Hexane.
4. The Technical Chlordane stock standard is purchased certified to contain 100ug/mL. A working standard of 400 ug/L is prepared by spiking 200uL of the stock standard and diluting to 50mL in Hexane.
5. Surrogate stock standard is purchased certified and contains Decachlorobiphenyl and Tetrachloro-m-xylene at 400ug/mL. Dilute 25uL of stock standard to 50mL in Hexane to make a 200ug/L solution. All prepared QC and samples are already spiked, and no additional surrogates are added. The analytical lab must spike all calibration standards.
6. ICV Standard: Stock standard solution containing all analytes of interest prepared from a source independent of the calibration standards. Dilute 3uL to 50mL with hexane to make a 60ug/L solution for the single component compounds. An ICV standard for Toxaphene and Chlordane is prepared when the analytes are requested.
7. Degradation (DEG) stock standard containing 4,4'-DDT and Endrin at 200ug/mL. A working standard of 80ug/L is prepared by spiking 20uL of the stock standard and diluting to 50mL in Hexane.
8. Standard Storage: Stock Standards can be stored for up to one year. All other working standards can be kept for up to 6 months or until stock standard it was made from expires, whichever is sooner. Standards must be replaced sooner if suspected of degrading or

concentrating. All standards shall be kept at 4°C when not in use. Store standards in amber glass with PTFE-lined caps and protect from light.

Preparing Calibration Standards:

1. The calibration standards are prepared by diluting stock solutions into 6 working levels. The following chart indicates the compounds and concentrations.

Table 1 Concentrations (µg/L)

	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6
ALPHA-BHC	2.5	5	20	60	80	100
GAMMA-BHC	2.5	5	20	60	80	100
4,4'-DDT	2.5	10	40	120	160	200
4,4'-DDD	2.5	10	40	120	160	200
DIELDRIN	2.5	10	40	120	160	200
ENDOSULFAN I	2.5	5	20	60	80	100
ENDRIN	2.5	10	40	120	160	200
HEPTACHLOR	2.5	5	20	60	80	100
METHOXYCHLOR	2.5	50	200	600	800	1000
ALDRIN	2.5	5	20	60	80	100
BETA-BHC	2.5	5	20	60	80	100
DELTA-BHC	2.5	5	20	60	80	100
α-CHLORDANE	2.5	5	20	60	80	100
γ-CHLORDANE	2.5	5	20	60	80	100
4,4'-DDE	2.5	10	40	120	160	200
ENDOSULFAN II	2.5	10	40	120	160	200
ENDOSULFAN SULFATE	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
HEPTACHLOR EPOXIDE	2.5	5	20	60	80	100
TOXAPHENE*	-	-	-	-	-	5000
TECHNICAL CHLORDANE*	-	-	-	-	-	400

2. *Multi-component compounds require a single calibration standard at the mid-level concentration.

RETENTION
TIME STUDY

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.
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.

DEGRADATION
STANDARD

1. A degradation check standard must be analyzed each day prior to analyzing samples and once every 12-hours the instrument continues to analyze samples. Inject 1 μ L of the 80ppb DEG working standard.
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.
3. The breakdown standard insures the instrument is maintained properly and will not cause breakdown of DDT and Endrin.
4. The degradation of either 4,4-DDT or Endrin cannot exceed 15% individually.

Breakdown DDT =

$$\frac{A_c + A_d}{A_t + A_c + A_d} \times 100$$

A_t = Peak Area of DDT
 A_c = Peak Area of DDE
 A_d = Peak Area of DDD

Breakdown of Endrin =

$$\frac{A_a + A_k}{A_c + A_a + A_k} \times 100$$

A_c = Peak Area of Endrin
 A_a = Peak Area of Endrin Aldehyde
 A_k = Peak Area of Endrin Ketone

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.

INITIAL CALIBRATION

The initial calibration consists of a series of standards analyzed at the concentration noted in Table 1. An initial calibration curve for each target analyte must be analyzed and evaluated before any peak for that analyte can be quantitated.

1. The calibration range is defined as the on-column concentration range adjusted for sample prep. 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 ug/mL). Typically, alpha-BHC is the only compound reported in the Level 1 standard:
 - a) At least five contiguous points must be used.
 - b) Calibration acceptance criteria must be met as described below.
 - c) The lowest calibration point must support the lowest reporting limit needed in the associated samples.
 - d) The QC spike amount must be within the calibration range.
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.
3. The calibration curve is now ready for analysis.
4. The acceptance criteria for initial calibration must be satisfied before analysis of samples begin. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.
5. Additionally one of the following options must be met. Always attempt to meet calibration criteria using the average response factor. If the average response factor does not pass, options B and C 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). The calibration options and requirements are as follows:
 - A. 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 all 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.

- B. 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 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.
- C. 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.
6. Determine the response factor for a multicomponent compound by calculating the average area count of the four major peaks. For Technical Chlordane and Toxaphene, the analyst should choose four peaks from the pattern for use in quantitation. These peaks should be chosen as discrete peaks and ones that have a sufficient response in the pattern. These same four peaks will be used for quantitation of all samples.

INITIAL CALIBRATION VERIFICATION

Immediately following the initial calibration procedure or before sample analysis, the analyst shall perform initial calibration verification (ICV). This will consist of a solution containing all target analytes prepared from a standard that is independent from the initial calibration.

The ICV must be analyzed following the initial calibration.

The ICV recovery must be 85-115%. Specific project criteria may apply that must be achieved for the associated samples. See SOP GEN-019.

If any target analyte recovery is outside the control limits, 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.

CONTINUING CALIBRATION
VERIFICATION
STANDARDS

1. The calibration verification standards are the mid-level standard of the six-point calibration. The calibration verification standards are analyzed to verify that the calibration curve and retention time windows are still valid.
2. The calibration verification standard is analyzed at the beginning of each 12-hour shift. The shift begins with the injection of the calibration verification standard and ends after the completion of analysis of the last sample or standard injected within 12 hours of the beginning of shift. A calibration verification standard must also be injected every 10 samples and at the end of the analytical sequence.
3. The criterion for the calibration verification standard is that all analytes must be ± 20 percent difference (%D) from the initial calibration. For each CCV, each compound must fall within the retention time window.
4. If the average of the responses for all analytes are within 20%, then the calibration has been verified; however, the % difference cannot exceed 30% for an individual analyte. Include the failure in the report narrative. This option is not allowed for some projects including, but not limited to, DOD, AFCEE, Marathon LLC, and projects from or reported to South Carolina. Check with the project manager if this option is used. See SOP GEN-019.
5. If the calibration verification standard fails to meet these criteria, repeat the injection of the standard. If the standard continues to fail, take appropriate corrective action (inspection of GC, re-prep, standard, etc.) If these criteria cannot be met, a new calibration curve shall be prepared.
6. If the calibration verification standard analyzed after a group of samples has a response for an analyte $>20\%$ 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. If an analyte was not

detected in the sample and the standard response is more than 20% below the initial calibration response, then re-injection is necessary.

SAMPLE ANALYSIS

1. Up to 10 samples (not including MB, LCS/LCSD) can be analyzed after the calibration verification standard.
2. All samples are evaluated on a primary column then a secondary column if an analyte above detection limit was detected. The secondary column confirms the presence of that analyte.
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. (The lower value maybe reported if required by the project.) If the difference between the two results, calculated as an RPD, is greater than 40%, narrate the sample results. The results are flagged in the report.

SAMPLE
RE-EXTRACTION

Samples are to be re-extracted due to failed QC or due to the sample results. When a MB, LCS, or LCSD fails to meet criteria, the entire batch is sent to extractions for re-extraction. If the MS, MSD, or surrogates fail criteria, only the affected samples are sent to extractions. Particular samples may be re-extracted if the sample results do not match historical values, if a sample and a duplicate do not match, or if physical differences are noted in samples. If samples are re-extracted outside of method specified holding times, both analyses are reported. To request sample/batch re-extraction, do the following:

1. If the extract has been analyzed, process the file and load to LIMS.
2. Enter the code "RP" into the analysis code; this will schedule new sample prep.
3. Complete the Re-extraction Request and Tracking Form (attached) and submit a copy to extraction supervisor.
4. When new extracts are brought to the lab, complete the original Re-extraction Request and Tracking Form and report the data appropriately.
5. If additional sample is not available to re-prepare, check with project manager to determine appropriate action to report the available data.
6. If two sets of data will be reported, see login to obtain a re-extracted sample number.

CALCULATION

1. $MS \% REC = \frac{MS \text{ Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$
2. $MSD \% REC = \frac{MSD \text{ Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$

$$3. \% \text{ RPD} = \frac{\text{MS} - \text{MSD}}{(\text{MS} + \text{MSD})} \times 100$$

LCS/LCSD results are substituted to calculate %RPD between LCS and LCSD. Results are calculated using the concentration (not percent recovery).

$$4. \text{ Response Factor} = A_s / C_s$$

A_s = Peak Area of analyte or surrogate
 C_s = Concentration of the analyte or surrogate

$$5. \text{ Surrogate/LCS Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

$$6. \text{ Concentration using RF:} \\ \text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(D)(V_t)}{(\text{RF})(V_s)}$$

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(D)(V_t)}{(\text{RF})(W_s)}$$

A_s = Area of peak for the analyte in sample
 D = Dilution factor
 RF = Mean Response factor from initial calibration
 W_s = Weight of sample extracted in g
 V_t = Total volume of concentrated extract in μL
 V_s = Volume of aqueous sample extracted in mL

7. Concentration using linear curve fit:

$$\text{Concentration } (\mu\text{g/L}) = [m(A_s) + b]D$$

$$\text{Concentration } (\text{mg/kg}) = [m(A_s) + b]D(5/W_s)$$

m = Inverse of slope
 A_s = Area of peak for the analyte in sample
 b = Intercept of the y-axis
 D = Dilution factor
 W_s = Weight of sample extracted

8. Concentration using a quadratic curve fit:

$$\text{Concentration } (\text{mg/L}) = [b + a(A_s) + a_2(A_s)^2]D(5/V_s)$$

$$\text{Concentration } (\text{mg/kg}) = [b + a(A_s) + a_2(A_s)^2]D(5/W_s)$$

$$9. \% \text{ Difference} = [(RF_I - RF_C) / RF_I] \times 100$$

RF_I = Average response factor from initial calibration
 RF_C = Response factor from current verification check standard

$$10. \% \text{ Drift} = \frac{(\text{Calculated Conc.} - \text{Theoretical Conc.})}{\text{Theoretical Conc.}} \times 100$$

$$11. \% \text{ RSD} = (\text{SD}/X) \times 100$$

RSD = Relative Standard Deviation
X = mean of initial RF's for a compound
SD = Standard Deviation of average RF's for a compound

QUALITY CONTROL

METHOD BLANK

1. The method blank (extraction blank) is analyzed to demonstrate the extraction procedure did not introduce contamination.
2. No target analytes should be detected in the method blank above $\frac{1}{2}$ the reporting limit. If any target analytes are detected, data may not be reported and samples must be re-extracted and re-analyzed unless the following apply:
 - A. If a target analyte is detected above $\frac{1}{2}$ the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
 - B. If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.
 - C. Additional project criteria may apply. See SOP GEN-019.

SURROGATES

1. The surrogate is used 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 Decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachloro-m-xylene (TCMX).
2. If a surrogate recovery fails below the lower control limit, re-extract and re-analyze the sample. If the surrogate is outside QC limits in the re-extract sample, then indicate in the case narrative and state that the recovery was outside the control limits due to sample matrix. If re-extraction cannot be performed due to insufficient sample, report the data with a narrative.
3. If the surrogate recovery is above the control limits and the sample results are less than the reporting limit, the data may be reported with a narrative.

<u>Surrogate</u>	<u>Rec. Limits % Water</u>	<u>Rec. Limits % Soil</u>
TCMX	45-148%	52-151
DCB	34-135%	56-159

4. Project specific limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

LABORATORY CONTROL
STANDARD (LCS/LCSD)

1. A LCS and LCSD (if required) are included in each batch to demonstrate the system is in control. Routinely the LCS/LCSD will be spiked with a full list of the analytes.
2. All recoveries should be within the control limits. If the full list of target analytes is spiked, a small percentage of sporadic failures will be allowed. See Table 2 for the number of allowable failures. The failures are noted in the case narrative. If a recovery is above the upper control limit and the sample results are below the reporting limit, the data may be reported with a narrative. For South Carolina projects, LCS/LCSD recoveries must be 70-130% to report data. For South Carolina, no sporadic marginal failures are allowed.
3. If a recovery is below the lower control limit or precision fails, the batch must be re-extracted and re-analyzed. If a re-extraction is not possible due to insufficient sample volume, report the data with a narrative.

TABLE 2: Number of Allowable Failures

Number of Analytes	Failures Allowed
0-11	0
11-30	1

4. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

MATRIX SPIKES (MS/MSD)

1. The purpose of the MS/MSD is to assess the performance of the method for a particular sample matrix. The recoveries for each spike compound should be within the control limits specified in Table 3.
2. Whenever the MS and/or MSD recoveries are outside the control limits, review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
3. If recoveries for the MS and/or MSD are outside the control limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.
4. If precision is outside of control limits, re-extract the parent, MS, and MSD. If the failure is repeated, check that the samples are homogenous. Narrate the sample results including corrective action and sample

appearance if applicable.

5. 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.
6. Spikes may be diluted out in the analysis process. Document this in the case narrative.
7. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

REPORTING LIMIT

Table 3 lists default-reporting limits for Pesticides. Reporting limits may be lowered by altering the prep and, when allowable, calibrating lower. The reporting limit should be no lower than 2X the verified MDL. Some projects may have different criteria for setting reporting limits. Check SOP GEN-019 and with the project manager.

Table 3 Reporting Limits

COMPOUND	WATER (ug/L)	SOLID (ug/kg)
alpha-BHC	0.05	2
beta-BHC	0.05	2
delta-BHC	0.05	2
gamma-BHC	0.05	2
Heptachlor	0.05	2
Aldrin	0.05	2
Heptachlor Epoxide	0.05	2
Endosulfan I	0.05	2
Dieldrin	0.10	4
4,4'-DDE	0.10	4
Endrin	0.10	4
Endosulfan II	0.10	4
4,4'-DDD	0.10	4
Endosulfan Sulfate	0.10	4
4,4'-DDT	0.10	4
Methoxychlor	0.50	20
Endrin Ketone	0.10	4
Endrin Aldehyde	0.10	4
alpha-Chlordane	0.05	2
gamma-Chlordane	0.05	2
Toxaphene	5.00	200
Technical Chlordane	0.25	10

MANUAL INTEGRATIONS

Perform and document manual integrations as described in SOP QA-010.

METHOD PERFORMANCE	<ol style="list-style-type: none">1) The method detection limit (MDL) is defined in Chapter One of the 8081B method. A laboratory should develop its own matrix-specific MDLs using the guidance found in Chapter One of the 8081B method.2) The chromatographic separations in this method have been tested in a single laboratory by using clean hexane and liquid and solid waste extracts that were spiked with the test compounds at three concentrations. Single-operator precision, overall precision, and method accuracy were found to be related to the concentration of the compound and the type of matrix.3) This method has been applied in a variety of commercial laboratories for environmental and waste matrices. Performance data were obtained for a limited number of target analytes spiked into sewage sludge and Dichloroethene still-bottoms at high concentrations. These data are provided in Tables 9 and 10 of the 8081B method.4) The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures used.5) Tables 9 and 10 of the 8081B method contain precision and recovery data generated for sewage sludge and Dichloroethane still-bottoms. Table 11 of the 8081B method contains recovery data for a clay soil, taken from Reference 10. The spiking concentration was for the clay soil was 500 ug/kg. The spiking solution mixed into the soil and then immediately transferred to the extraction device and immersed in the extraction solvent. The spiked sample was then extracted by Method 3541 (Automated Soxhlet). The data represent a single determination. Analysis was by capillary column gas chromatography/electron capture detector following Method 8081 for the Organochlorine Pesticides.
POLLUTION PREVENTION	See QAPP Section 13.2
WASTE MANAGEMENT	See SOP GEN-009
IDOC	See SOP QA-014
MDL	See SOP QA-009

GULF COAST ANALYTICAL LABS
GC - PCB METHOD 8082A
STANDARD OPERATING PROCEDURE

PROCEDURES: GC-023
PAGE: 1 OF 11
EFFECTIVE DATE: 10/07/2008
APPROVED BY: *MAP*
QA/QC APPROVED: *JDT*

SUBJECT

SCOPE AND APPLICATION

This method provides gas chromatographic procedures for the detection of ppb levels of certain polychlorinated biphenyls (PCB'S). Prior to the use of this method, appropriate extraction techniques must be used. The extraction is described in EXT-002 for solid samples and EXT-010 for aqueous samples. An aliquot of the sample extract is injected into a GC equipped with an ECD detector.

MATRIX

Water and Solid

REFERENCE

SW846 Method 8082A, 8000C
For North Carolina and South Carolina projects SW-846 8000B must be used instead of SW-846 8000C

PRESERVATIVE

Cool to 4°C

HOLDING TIME

Water - From collection to extraction - 7 days
From extraction to analysis - 40 days
Solid - From collection to extraction - 14 days
From extraction to analysis - 40 days

DEFINITIONS

See SOP GEN-016

SAFETY

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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

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.
INTERFERENCES	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.
INSTRUMENTATION	<p>Gas Chromatograph should be suitable for split-less or on-column injection. The system should be equipped with an electron capture detector. The instruments used are Agilent Technology 6890N GC/ECD.</p> <p>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.</p>
COLUMNS	<p>Recommended Columns are</p> <p>XLB or similar phase 30m X 0.32mm ID 35MS or similar phase 30m X 0.32mm ID</p>
REAGENTS	Hexane-pesticide grade. Store away from sources of Phthalates.
STANDARDS	<ol style="list-style-type: none">1. Purchasing Standards: All standards should be purchased from a certified vendor. The ICV shall be from a secondary source.2. ICV Standard: Stock standard solution containing Aroclor 1016 and Aroclor 1260 prepared from a source independent of the calibration standards.3. Surrogate stock standard is purchased certified and contains decachlorobiphenyl and Tetrachloro-m-xylene at 400ug/mL. Dilute 25uL of stock standard to 50mL in Hexane to make a 200ug/L solution. All prepared QC and samples are already spiked, and no additional surrogates are added. The analytical lab must spike all calibration standards.4. Standard Storage: Stock Standards can be stored for up to one year. All other working standards can be kept for up to 6 months or until stock standard it was made from expires, whichever is sooner. Standards must be replaced sooner if suspected of degrading or concentrating. All standards shall be kept at 4°C when not in use. Store standards in amber glass with PTFE-lined caps and

protect from light.

Preparing Calibration Standards:

1. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 is diluted into six working levels.

Table 1 Concentrations (ug/L)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Aroclor 1016	50	100	200	400	800	1600
Aroclor 1260	50	100	200	400	800	1600

2. The remaining five Aroclors (Aroclor 1221, Aroclor 1232, Aroclor 1242, Aroclor 1248, Aroclor 1254) are calibrated using one working level of each. The concentrations of each are equivalent to the concentration of Level 3 of the Aroclor 1016/1260 mix.

RETENTION
TIME STUDY

1. For all individual components 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.
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.

INITIAL CALIBRATION
CURVE

The initial calibration consists of a series of six levels of Aroclor 1016/1260 mix and one mid-level of each of the remaining five Aroclors. An initial calibration curve for each target analyte must be analyzed and evaluated before any result for that analyte can be quantitated.

Average Response Factor Calibration.

1. For each of the six levels of Aroclor 1016/1260, calculate the response factor of 5 major characteristic Aroclor peaks in each of the initial calibration

standards. These peaks should be chosen as discrete peaks present in each Aroclor, which would not be subject to interference from other Aroclors. Each peak chosen should have a sufficient response to allow for accurate quantitation of the detected Aroclors. The analyst should be aware of the affect of weathering on Aroclor patterns and careful consideration should be given to patterns detected in samples when choosing peaks.

2. For each Aroclor, 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.
3. For each of the single-point midlevels of the remaining Aroclors, calculate the response factor for five of the characteristic Aroclor peak in each single-point.
4. 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 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.
5. 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.

6. 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.

INITIAL CALIBRATION
VERIFICATION

1. Immediately following the initial calibration procedure, the analyst will perform an initial calibration verification (ICV). This will consist of a solution containing Aroclor 1016 and 1260 prepared from a standard that is independent from the standards used for the initial calibration.
2. The ICV must be analyzed following the initial calibration. The ICV recovery must be 85-115% recovery.
3. If any target analyte exceed(s) the recovery criteria, corrective action must be taken. This may include instrument maintenance, re-analysis of ICV or initial calibration, or re-preparation of the standards involved.

CONTINUING CALIBRATION
VERIFICATION
STANDARDS

1. The calibration verification standard is a mid-level of the Aroclor 1016/1260 mix. The calibration verification standard is analyzed to verify that the calibration curve and retention time windows are still valid.
2. The calibration verification standard is analyzed at the beginning of each 12-hour shift. The shift begins with the injection of the calibration verification standard and ends after the completion of analysis of the last sample or standard injected within 12 hours of the beginning of shift. Calibration verification standard must also be injected every 10 samples and at the end of the sequence.
3. The criteria for the calibration verification standard is that all analytes must be <20 Percent Difference (%D) from the initial calibration. For each CCV, each compound must fall within the retention time window.
4. If the calibration verification standard fails to meet this criterion, repeat the injection of the standard. If the standard continues to fail, take appropriate

corrective action (inspection of GC, re-prep. standard, etc.) If this criteria cannot be met, a new initial calibration curve should be prepared.

6. If the calibration verification standard analyzed after a group of samples has a response for an analyte >20% 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. If an analyte was not detected in the sample and the standard response is more than 20% below the initial calibration response, then re-injection is necessary.

SAMPLE ANALYSIS

1. Up to 10 samples (not including MB, LCS/LCSD) can be analyzed after the calibration verification standard.
2. All samples are evaluated on a primary column then a secondary column if an analyte above detection limit was detected. The secondary column confirms the presence of that analyte.
3. If the analyte falls within the retention time window and the Aroclor pattern is verified on both the primary and secondary analysis, then calculate the concentration present. The higher value of the two analyses is reported. (The lower value maybe reported if required by the project.) If weathered peaks are present, they may fall outside the retention time window but be identified by pattern. This data would be reported with a narrative. If the difference between the two results, calculated as an RPD, is greater than 40%, narrate the sample result. The results are flagged in the report.

SAMPLE RE-EXTRACTION

Samples are to be re-extracted due to failed QC or due to the sample results. When a MB, LCS, or LCSD fails to meet criteria, the entire batch is sent to extractions for re-extraction. If the MS, MSD, or surrogates fail criteria, only the affected samples are sent to extractions. Particular samples may be re-extracted if the sample results do not match historical values, if a sample and a duplicate do not match, or if physical differences are noted in samples. If samples are re-extracted outside of method specified holding times, both analyses are reported. To request sample/batch re-extraction, do the following:

1. If the extract has been analyzed, process the file and load to LIMS.
2. Enter the code "RP" into the analysis code; this will

- schedule new sample prep.
3. Complete the Re-extraction Request and Tracking Form (attached) and submit a copy to extraction supervisor.
 4. When new extracts are brought to the lab, complete the original Re-extraction Request and Tracking Form and report the data appropriately.
 5. If additional sample is not available to re-prep, check with project manager to determine appropriate action to report the available data.
 6. If two sets of data will be reported, see login to obtain a re-extracted sample number.

CALCULATION

1. MS % REC = $\frac{\text{MS Concentration}-\text{Sample Concentration}}{\text{spike added}} \times 100$
2. MSD % REC = $\frac{\text{MSD Concentration}-\text{Sample Concentration}}{\text{spike added}} \times 100$
3. % RPD = $\frac{\text{MS}-\text{MSD}}{(\text{MS}+\text{MSD})/2} \times 100$

LCS/LCSD results are substituted to calculate %RPD between LCS and LCSD. Results are calculated using the concentration (not percent recovery).

4. Response Factor = A_s/C_s
 A_s = Peak Area of analyte or surrogate
 C_s = Concentration of the analyte or surrogate
5. Surrogate/LCS Recovery = $\frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$
6. Concentration using RF:
 Concentration ($\mu\text{g/L}$) = $\frac{(A_s)(D)(V_t)}{(RF)(V_s)}$
 Concentration ($\mu\text{g/kg}$) = $\frac{(A_s)(D)(V_t)}{(RF)(W_s)}$

A_s = Area of peak for the analyte in sample
 D = Dilution factor
 RF = Mean Response factor from initial calibration
 W_s = Weight of sample extracted in g
 V_t = Total volume of concentrated extract in μL
 V_s = Volume of aqueous sample extracted in mL

7. Concentration using linear curve fit:
 Concentration ($\mu\text{g/L}$) = $[m(A_s)+b]D$
 Concentration (mg/kg) = $[m(A_s)+b]D(5/W_s)$
 m = Inverse of slope
 A_s = Area of peak for the analyte in sample

b = Intercept of the y-axis
D = Dilution factor
W_s = Weight of sample extracted

8. Concentration using a quadratic curve fit:

$$\text{Concentration (mg/L)} = [b + a(A_s) + a_2(A_s)^2]D(5/V_s)$$

$$\text{Concentration (mg/kg)} = [b + a(A_s) + a_2(A_s)^2]D(5/W_s)$$

9. % 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

10. % Drift = $(\text{Calculated Conc.} - \text{Theoretical Conc.}) / \text{Theoretical Conc.} \times 100$

11. % RSD = $(SD/X) \times 100$

RSD = Relative Standard Deviation
X = mean of initial RF's for a compound
SD = Standard Deviation of average RF's for a compound

QUALITY
CONTROL

METHOD BLANK

1. The method blank (extraction blank) is analyzed to demonstrate the extraction procedure did not introduce contamination.
2. No target analytes should be detected in the method blank above ½ the reporting limit. If any target analytes are detected, data may not be reported and samples must be re-extracted and re-analyzed unless the following apply.
 - A. If a target analyte is detected above ½ the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
 - B. If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.
 - C. Additional project criteria may apply. See SOP GEN-019.

SURROGATES

1. The surrogate is used to verify that each sample was properly extracted. A surrogate is a non-target compound

that is chemically similar to the analytes. The surrogate used is decachlorobiphenyl (DCB).

2. If a surrogate recovery fails below the lower control limit, re-extract and re-analyze the sample. If the surrogate is outside QC limits in the re-extract sample, then indicate in the case narrative and state that the recovery was outside the control limits due to sample matrix. If re-extraction cannot be performed due to insufficient sample, report the data with a narrative.
3. If the surrogate recovery is above the control limits and the sample results are less than the reporting limit, the data may be reported with a narrative.

<u>Surrogate</u>	<u>Rec. Limits % Water</u>	<u>Rec. Limits % Soil</u>
DCB	34-135%	56-159

4. Project specific limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

LABORATORY CONTROL STANDARD (LCS/LCSD)

1. A LCS and LCSD (if required) are included in each batch to demonstrate the system is in control. Routinely the LCS/LCSD will be spiked with a mixture of Aroclors 1016/1260. Spikes for specific Aroclors may be required for specific projects. Control limits are listed in the table below. For South Carolina projects, LCS/LCSD recoveries must be 70-130% to report data.

LCS/LCSD CONTROL LIMITS

COMPOUND	% RECOVERY WATER	RPD WATER	% RECOVERY SOLID	RPD SOLID
Aroclor 1016	55-131	35	62-124	31
Aroclor 1260	51-138	34	62-129	36

*Other Aroclors- based on project requirements

2. All recoveries must be within the control limits. If a recovery is above the upper control limit and the sample results are below the reporting limit, the data may be reported with a narrative.
3. If a recovery is below the lower control limit, the batch must be re-extracted and re-analyzed. If a re-extraction is not possible due to insufficient sample volume, report the data with a narrative.
4. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

MATRIX SPIKES (MS/MSD)

1. The purpose of the MS/MSD is to assess the performance of the method for a particular sample matrix. Use the same precision and accuracy control limits listed for the LCS/LCSD.
2. Whenever the MS and/or MSD recoveries are outside the control limits, review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
3. If recoveries for both the MS/MSD are outside the control limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.
4. If precision is outside of control limits, re-extract the parent, MS, and MSD. If the failure is repeated, check that the samples are homogenous. Narrate the sample results including corrective action and sample appearance if applicable.
5. 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.
6. Spikes may be diluted out in the analysis process. Document this in the case narrative.
7. Project specific recovery and precision limits may apply and may be more stringent; project limits are listed in the LIMS. See SOP GEN-019.

REPORTING LIMITS

Below is a list of the default reporting limits for PCBs. Reporting limits may be lowered by altering the prep and, when allowable, calibrating lower. The reporting limit should be no lower than 2X the verified MDL. Some projects may have different criteria for setting reporting limits. Check SOP GEN-019 and with the project manager.

Water - 1 ug/L
Solid - 40-1200 ug/kg

METHOD PERFORMANCE

1. The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and calibration procedures

used. Table 9 of Method 8082 provides single laboratory recovery data for Aroclors spiked into clay and soil and extracted with automated Soxhlet. Table 10 of Method 8082 provides multiple laboratory data on the precision and accuracy for Aroclors spiked into soil and extracted by automated Soxhlet.

2. During method performance studies, the concentrations determined as Aroclors were larger than those obtained using the congener method. In certain soils, interference prevented the measurement of congener 66. Recoveries of congeners from soils spiked from Aroclor 1254 and Aroclor 1260 were between 80% and 90%. Recoveries of congeners from environmental reference materials ranged from 51-66% of the certified Aroclor values.

MANUAL INTEGRATIONS	Perform and document manual integrations as described in SOP QA-010.
POLLUTION PREVENTION	See QAPP Section 13.2
WASTE MANAGEMENT	See SOP GEN-009
IDOC	See SOP QA-014
MDL	See SOP QA-009

GULF COAST ANALYTICAL LABORATORIES, INC
GCMS - VOLATILES
STANDARD OPERATING PROCEDUREPROCEDURES: GCMSV-003
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EFFECTIVE DATE: 5/4/09
APPROVED BY: *MAP*
QA/QC APPROVED: *JST*

SUBJECT SCOPE AND APPLICATION

This is a purge and trap GC/MS method, which quantitatively identifies purgeable organic compounds. A list of target analytes is available in the LIMS.

A 5-mL water sample is purged, with helium 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 helium to desorb the purgeable compounds onto the column. The compounds are then separated by their molecular weight, the column phasing and increasing temperature, and then detected by a mass spectrometer.

MATRIX Water and Solids

REFERENCE SW846 Methods 8260B, 8260C, 5030B, 5030C, 5035A, 8000B, 8000C
NOTE: For North Carolina and South Carolina projects, Method SW846 8260B and 8000B must be used.PRESERVATIVE Cool <6°C Aqueous pH <2 with HCl and/or Na₂S₂O₃ (unless unpreserved requested)
Acrolein and Acrylonitrile pH 4-5 with Na₂S₂O₃, if unpreserved holding time is 3 days.HOLDING TIME Water - 14 days from sample collection when preserved in HCl
Solid - 14 days from sample collection
Solid - Encore™ Samplers - 48 hours to preservation/then 14 days to analysis

Specific projects may require non-acid preservation of water samples. If water samples are unpreserved the analysis must be performed in 7 days.

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, narrate results.

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and the supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins,

carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures, solvents and chemicals must be handled in ventilated hoods.

INTERFERENCES

Contamination can be related to impurities in the purge gas and solvent vapors in the lab. The system is shown to be free from contamination by running a reagent blank each 12-hour period.

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 C22, C30, and C4.

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 until an acceptable blank is achieved and/or clean the purge vessel and bake trap. Reanalyze samples with potential carryover contamination.

INSTRUMENTATION & APPARATUS

Agilent 6890 and 7890 GC with a 5973 or 5975 Mass Spectrometer coupled with a Teledyne Tekmar purge and trap and autosampler

HP5890 GC with 5971/5972 Mass Spectrometer coupled with a Velocity XPT concentrator using a Solatek 72 or
Each instrument is assigned a maintenance logbook. All maintenance performed on the instrument must be recorded.

Target data acquisition system

DB-624 column

Gas-tight syringes

Electronic Balance - capable of weighing to 0.01g

Volumetric flasks

44 mL VOA vials

2 mL autosampler vials

Glass culture tubes

Pasteur pipets

Magnetic stir bars

Scoopula spatulas

pH paper

REAGENTS

All organic solvent shall be of pesticide grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.

Water

Methanol - purge and trap reagent grade or equivalent

Blank sand

Sodium bisulfate

STANDARDS

All standards used must be pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

The following describes the standards used and their preparation:

1. All stock standards are received in glass sealed ampules.
- A. The following stock standards are used, as necessary to prepare solutions for calibration, CCV, LCS, and MS samples:
 - A.1 Stock standard solutions - 1000mg/L (8260 mix) - commercially prepared certified solutions in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.2 Stock standard solutions - 2000mg/L (ketone mix) - commercially prepared certified solutions in 4:1 methanol/water, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ until the documented manufacturer's expiration date.
 - A.3 Stock standard - 2000 mg/L custom gas mix - commercially prepared certified solution in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.4 Stock standard - Acrolein/Acrylonitrile (A-2000 mg/L; Vinyl Acetate - 400 mg/L) (AAV) - commercially prepared certified solution in water, stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.5 Stock standard - 2-Chloroethyl vinyl ether (CVE) - 1000 mg/L - commercially prepared certified solutions in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.6 Stock standard - Appendix IX compounds - 3 mixes - 2500/200 mg/L - commercially prepared certified solutions in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.7 Stock standard solutions - oxygenate mix (2000 mg/L; t-Amyl Alcohol and Ethanol 8000mg/L) - commercially prepared certified solutions in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date.
 - A.8 Additional standards may be run that are project specific. All standards shall be certified and stored according to the manufacturer's instructions.

- B. Independent stock standards - standard mixes identical to those listed above from a secondary source or a secondary lot number from the same manufacturer. These standards are used to prepare the ICVs.
- C. Stock Surrogate mix - 2500 mg/L (SS)- commercially prepared certified solution in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$, until the documented manufacturer's expiration date. This solution is also used to prepare BFB tune check solution.
- D. Stock Internal Standard mix - 2500 mg/L (IS) - commercially prepared certified solution in methanol, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ until the documented manufacturer's expiration date.
2. Secondary standards - Secondary standards are prepared by diluting the stock standard as described below. The three standards, listed in Table 1, labeled 8260 are prepared together, then transferred to a vial and stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$. All other standards are stored in vials with minimum headspace or flame sealed and may be held for 6 months or until manufacturer's expiration, whichever is sooner.
Procedures for volatile standard preparation:
 - A. Assemble the necessary glassware, syringes and solvent. Purge and trap reagent grade methanol shall be used for all VOA standard preparation.
 - B. Ensure that all glassware and syringes are clean and free of moisture by rinsing with at least three aliquots of methanol. Volumetric glassware should never be stored in or placed in heated ovens. The use of dedicated glass will help prevent cross-contamination of standards.
 - C. Fill the volumetric glassware to approximately 75 percent volume. Addition of standards shall proceed by adding the more highly volatile compounds last to prevent compound loss.
 - D. Add the proper volume of standards using a syringe. Minimum agitation should occur upon adding the standard solution.
 - E. After the addition of standard, the volumetric flask should be diluted until the meniscus reaches the calibration mark.
 - F. Insert the glass stopper and mix by inverting three times.
 - G. Cool the flask and its contents to approximately 5°C .
 - H. Using a Pasteur pipette, transfer the standard to a 2 mL ampule. Seal the ampule immediately.
 - I. Standards may be stored in mininert vials with minimal headspace.
 - J. Label the vial with the following information:
 - Standard Name
 - Standard ID #
 - Initials of Person Preparing Sample
 - Date of Preparation
 - Expiration Date
 - K. Store the vial the same as the stock standard.

- L. Dispose of excess materials in accordance with established laboratory procedures.
- M. Always quantitate new standards against known standards to ensure accurate concentration levels.
- N. Fill out the standards logbook in accordance with proper laboratory procedure and enter required information in LIMS.
- O. A combined solution containing the internal standards and surrogates is prepared as the secondary standard. IS/SS is added automatically by the Solatek autosampler.
- O.1 Internal standards - The recommended internal standards are Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Each calibration standard, blank, LCS, and sample undergoing GC/MS analysis must be spiked with 50 ug/L of the internal standard spiking solution prior to analysis. Different internal standards may be used as necessary.
- O.2 Surrogate standards - The recommended surrogates are Toluene d-8, Bromofluorobenzene, 1,2 - Dichloroethane-d4, and Dibromofluoromethane. Each, blank, LCS, and sample undergoing GC/MS analysis should be spiked with 50 ug/L of the surrogate spiking solution prior to analysis. Different surrogate standards may be used as necessary.

Table 1
Secondary Standards

	Stock Conc.	Initial Vol.	Final Vol.	Final Conc.	Solvent
8260 mix	1000 mg/L	6.25 mL	25 mL	250 mg/L	Methanol
8260 gases	2000 mg/L	3.125 mL	25 mL	250 mg/L	Methanol
8260 Ketones	2000 mg/L	3.125 mL	25 mL	250 mg/L	Methanol
CVE	1000 mg/L	6.25 mL	25 mL	250 mg/L	Methanol
AAV	2000/400 mg/L	3.125	25 mL	250/50 mg/L	Deionized water
APP IX Mix 1 & 3	2500 mg/L	2.5 mL	25 mL	250 mg/L	Methanol
APP IX Mix 2	200 mg/L	2.5 mL	10 mL	50 mg/L	Methanol
Oxygenate	2000/8000 mg/L	1.25 mL	10mL	250 mg/L	Methanol
SS	2500 mg/L	1.0 mL	50 mL	50 mg/L	Methanol
IS	2500 mg/L	1.0 mL	50 ml	50 mg/L	Methanol
IS/SS	2500 mg/L	1.0 mL of each	50 mL	50 mg/L	Methanol

3. The following working standards are prepared from the secondary standards and diluted in reagent water:
- A. Calibration standards are prepared at a minimum of five concentrations for all target compounds and surrogates (additional concentrations may be analyzed) from the secondary dilution of stock standards. Prepare these standards in deionized water. The calibration standards are

prepared immediately before use at concentrations of 5, 20, 50, 100, and 200 ug/L for solids and waters using a 5mL purge. For waters using a 25mL purge prepare the standards at concentrations of 1, 2, 5, 10, and 25 ug/L. Each standard is prepared in a volumetric flask or a 5ml gas tight syringe. Lower concentration standards may be required to meet project requirements. The 8260 mix, ketones, gases, CVE, AAV, and surrogate standards (250 ug/L) are combined. The three APP IX mixes are also combined and are calibrated separately. Analysis of analytes not contained in any of the aforementioned standards may be calibrated for in combination with the 8260 mix, ketones, gases, CVE, and AAV if sufficient resolution is achieved, or may be calibrated for separately. 44mL of each standard is transferred to a 44mL VOA vial for water calibrations performed using the Solatek 72 autosampler. 5mL of each standard is transferred to a 44mL low bleed septa vial for solid calibrations.

- B. Initial calibration verification standard - Prepare a 50 ug/L standard from the secondary dilution of the independent stock standard (250/50 mg/L). The mixes are combined in the same manner as the calibration standards.
- C. LCS and Matrix Spiking Standards - spike at a concentration of 10 ug/L (25 mL purge) and 50 ug/L (5 mL purge and solids) using the working standards.

RECOMMENDED
CONDITIONS

The following list conditions that may be used in the lab. The analyst is allowed to modify settings to optimize operating conditions.

GC Instrument Conditions

Oven Temperature: 35°C
Rate A: 25°C/min to 180°C
Rate B: 45°C/min to 245°C
Hold: 2.26 min

Purge & Trap Conditions

Purge: 11 minutes at 30°C
Desorb Preheat: 205°C
Desorb: 1.5 minutes at 250°C
Bake: 6.5 minutes at 260°C

PROCEDURE

1. Procedures for instrument tuning
 - A. The instrument should be tuned using FC-43 (perfluorotributylamine) such that all tuning criteria for BFB can be met (Table 2). Before analysis begins, each instrument must be tuned checked by direct injecting 2 uL of a solution containing 25 mg/L BFB or by purging an amount equivalent to 50ng of BFB standard.
 - B. The mass spectrum of 4-Bromofluorobenzene (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 fails.

- C. 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 is still not met instrument maintenance is performed.
- D. 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.
- E. 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.

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

2. Procedures for initial calibration

- A. The initial calibration standards are prepared at a minimum of five different concentrations from the secondary dilution. Additional standards are usually included for specific compounds including a 0.2ppb, 0.4ppb, 1ppb, 2ppb, and 10ppb.
The calibration range is defined as the on-column concentration range.
 - 1. At least five contiguous points must be used (six if a quadratic curve fit is employed).
 - 2. Calibration acceptance criteria must be met as described below.
 - 3. The lowest calibration point must support the lowest reporting limit needed in the associated samples.
 - 4. The QC spike amount must be within the calibration range.
- B. The standards are analyzed in the following sequence:
 - B.1. BFB (50 NG)
 - B.2. 5 PPB initial calibration
 - B.3. 20 PPB initial calibration
 - B.4. 50 PPB initial calibration
 - B.5. 100 PPB initial calibration
 - B.6. 200 PPB initial calibration

Note: 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.

- C. Once the five levels of standards have been analyzed, the validity of the initial calibration must be verified by comparing the SPCCs and CCC's to the following criteria.
- C.1. Calibration check compounds (CCC) listed below must be $\leq 30\%$ RSD.

<u>CCC's</u>	<u>% RSD</u>
1,1-Dichloroethene	<30%
Chloroform	<30%
1,2-Dichloropropane	<30%
Toluene	<30%
Ethylbenzene	<30%
Vinyl Chloride	<30%

- C.2. System Performance Check Compounds (SPCC) must have a minimum (RF) as indicated. All SPCC's must be evaluated at each level of the calibration curve to insure that compound instability and degradation are checked as required by the method.

<u>SPCC's</u>	<u>MINIMUM RF</u>
Chloromethane	0.1
1,1-Dichloroethane	0.1
Bromoform	0.1
1,1,2,2-Tetrachloroethane	0.3
Chlorobenzene	0.3

- D. Additionally one of the following options must meet criteria. The analyst will judge criteria in the order given or with prior knowledge of the behavior of target compounds. One of the following must pass before a calibration is considered acceptable:
- D.1 All target analytes have an RSD less than 15%.
- D.2 A first order least square regression may be used if $r^2 \geq 0.995$. 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 cannot be used.
- D.3 A quadratic curve fit is used with 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. The analyst should check the y-intercept (b). If the intercept is greater than half the reporting limit, this option cannot be used. The quadratic calibration option is not allowed for South Carolina projects.
- D.4 Alternatively, if the analyte is listed as a poor performer and does not meet the criteria listed above, the data may be reported with a narrative if authorized by the client. NOTE: Compounds cannot be classified as poor performers for South Carolina projects.
- E. If the initial calibration does not conform to the above listed criteria, corrective action must be taken.
- F. The position of the retention time should be set using the mid-point of an initial calibration, and must be checked and reset as necessary after source cleaning or column maintenance.

3. Initial Calibration Verification
 - A. Immediately following the initial calibration procedure or before sample analysis, the analyst shall perform initial calibration verification (ICV). This will consist of a solution containing all target analytes prepared from a standard that is independent (second source) from the standards used for the initial calibration.
 - B. The ICV must be analyzed following the initial calibration. The ICV must exhibit a recovery of 70-130%, except for the targets listed in Table 3, which must be recovered between 60-140%. Recovery requirements may differ on a project specific basis. See SOP GEN-019. All analytes must exhibit a recovery of 70-130% for South Carolina samples.
 - C. If any target analyte recovery is outside the control limits, corrective action must be taken (project specific exceptions may apply). This may include instrument maintenance, re-analysis of the ICV or initial calibration, or re-preparation of the standards involved. If the target fails high and is not present in the associated samples, the data may be reported with a narrative.
4. Procedures for Continuing Calibration
 - A. The 50 PPB continuing calibration is analyzed in each batch after acceptable BFB tune and before samples. The following criteria shall be achieved before sample analysis:
 - A.1 CCC's must meet the specified criteria for all target analytes. Calibration Check Compounds (CCC's) are allowed no greater than 20% difference from the initial calibration if calibration option 2.D.1 or 2.D.4 is used or % drift $\leq 20\%$ if a linear or quadratic calibration option was used. The quadratic calibration option is not allowed for South Carolina projects.
 - A.2 System performance-check compounds (SPCC's) are required to maintain a relative response factor (RF) as indicated in 2C.2.
 - A.3 All target analytes should have a %Difference $< 20\%$ from the initial calibration if calibration options 2.D.1 or 2.D.4 were used or %Drift $\leq 20\%$ of expected value if a linear or quadratic calibration option was used. All targets must have difference/drift $\leq 30\%$. Project specific limits may apply and are listed in the LIMS. The quadratic calibration option is not allowed for South Carolina projects.
 - A.4 Some compounds have been classified as poor performers due to poor purging efficiency. These analytes are listed in Table 3 and are acceptable at $\leq 40\%$ difference/drift. Poor performer exceptions are not allowed for South Carolina samples.

Table 3
Targets that exhibit poor purging efficiency

Acetone	Ethyl acetate
Acetonitrile	2-hexanone
Acrolein	Isobutyl alcohol
Acrylonitrile	Methacrylonitrile
2-Butanone	4-methyl-2-pentanone
t-butyl alcohol	Pentachloroethane
1,2-dibromo-3chloropropane	Propionitrile

1,4-dichloro-2-butene	Benzyl chloride
1,4-dioxane	1-Nitropropene
2-Chloropropene	1,3 Butadiene
Isopropyl ether	Tetrahydrofuran
2-Nitropropane	2,2,4-Trimethylpentane
2-Chloroethylvinyl ether	Ethyl ether
2-Butanol	Ethyl methacrylate
Cyclohexanone	Dicyclopentadiene
3-Dichloro-1-propene	n-Butanol
Allyl chloride	Methyl methacrylate
Chloroprene	Dichlorodifluoromethane
Bromomethane	TBA
3,3-Dimethylbutanol	t-Amyl Alcohol
Ethanol	

- A.5 If the target analyte (not a CCC) has failed, the RF has increased, and there are no hits during sample analysis, the data is reportable with a narrative. Note: no failures are allowed for South Carolina projects.
- A.6 The retention time for any internal standard shall not vary any more than 30 seconds from the mid-point standard of the most recent initial calibration.
- A.7 The internal standard area must not deviate by more than a factor of two (-50% to + 100%) from the mid-point standard of the most recent initial calibration.
- A.8 Project specific criteria may apply and may be more stringent. See SOP GEN-019.
- B. If specified criteria are not met, corrective action must be taken. If no source of the problem can be determined after corrective action has been taken, a new initial calibration must be generated.
5. The purpose of the method blank is to ensure that the analytical system is free of contamination and carryover. The method blank is run at least once per batch (not to exceed 20 samples) before sample analysis. The method blank shall be handled in exactly the same manner as the samples that it represents.
- A. The following are a description of the three types of method blanks commonly used in routine analysis and the analysis and evaluation of storage blanks.
- A.1 Water - Water blanks require no special preparation. Fill a VOA vial with reagent water. Place the full 44 mL VOA vial on the SOLATEk autosampler. 5 uL of the internal standard/surrogate standard will be added automatically.
- A.2 Low-level soil - Low-level soil method blanks require special preparation techniques. Place the 5.0 grams of sand in a 44 mL vial and add 5.0 mL of deionized water. Place on the autosampler. The SOLATEk will add 5.0 mL of deionized water and the internal/surrogate standards.
- A.3 Medium/High level soil - medium/high level soil blanks are prepared by weighing 5.0 grams of sand into a 44 mL VOA vial. Add 5.0 mL Methanol. Shake the vial to extract any volatiles into the solvent. A 1000 uL extract aliquot is diluted to 50ml in a volumetric flask with deionized water. Place on the autosampler.

- The autosampler will add an amount of internal standard/ surrogate solution equivalent to 50ug/L. Document preparation of blank in logbook.
- A.4 Storage Blanks - A set of at least two storage blanks filled with reagent water are kept in each cooler used to store samples. Storage blanks are analyzed every two weeks. If there is detection above $\frac{1}{2}$ the reporting limit, analyze the second vial to confirm the detection. If the results are confirmed immediately notify QA of the affected blank, cooler tested, and storage time. All data must be filed in the appropriate logbook.
- B. To prove a contaminant free system, no target analytes shall be detected in the method blank above one half the reporting limit except for common laboratory contaminants, which shall be less than the reporting limit. If any target analytes are detected above this level, data shall not be reported and samples must be re-extracted and re-analyzed unless the following apply:
- B.1 If a target analyte is detected above the reporting limit, data may be reported if the concentration is not greater than 10% of the measured concentration in associated samples. Include a narrative with the data.
- B.2 If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.
- B.3 If re-analysis is not possible, flag the associated samples and include a narrative in the report. Document the action taken.
- C. The source of blank contamination shall be investigated and measures taken to eliminate future contamination.
- D. Project specific criteria may apply and may be more stringent. See SOP GEN-019.
6. Procedure for Laboratory Control Spike (LCS/LCSD). An LCS and LCSD are included in each batch of up to 20 samples to demonstrate the system is in control. An LCSD is not required if an MS/MSD is performed. Routinely the LCS/LCSD will be spiked with the full list of 8260 analytes at 50ug/L. The CCV performed that day may be used as the LCS if allowed by the project. Check with the project manager for project specific spiking criteria; some projects may require all target analytes are spiked. The LCS is assessed using the following criteria:
- A. The percent recovery is calculated for all analytes spiked (see Calculation section). Recoveries are compared to the appropriate control limits. In-house control limits are used if project specific limits have not been requested, and are available in the LIMS. Project specific control limits may be requested; check with the project manager for limits needed.
- B. If a recovery is above the upper control limit and the sample results are below the reporting limit, the data may be reported with a narrative. All analytes must pass for South Carolina samples.
- C. If a recovery is below the lower control limit, the batch must be re-analyzed unless it meets the requirements of a sporadic failure (see below). If a re-extraction is not

possible due to insufficient sample volume, report the data with a narrative. All analytes must pass for South Carolina samples.

- D. If the full list of target analytes is spiked, a small number of sporadic failures will be allowed. The failure cannot include a primary compound of interest as defined by the client. See Table 4 for the number of allowable failures. The failures are noted in the case narrative. These failures shall be monitored to ensure randomness. This shall be defined as not exceeding failure in 1 of 3 LCS determinations. If the same analyte fails repeatedly and is not included in Table 3, locate the source of the problem and perform corrective action. All analytes must pass for South Carolina samples.

Table 4
Number of Allowable Failures

Number of Analytes	Failures Allowed
<11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

- E. If an LCS and an LCSD are analyzed, calculate precision using the RPD in the Calculation section, equation 2. Substitute the LCS and LCSD results for the MS/MSD. Precision is assessed using the criteria in the LIMS.
7. Procedures for Matrix Spikes
- A. Analyze one matrix spike/matrix spike duplicate (MS/MSD) pair with each batch of up to 20 samples processed together. The purpose of the spikes is to confirm the matrix being analyzed is not interfering with the recovery of the analytes.
- B. The MS/MSD spike includes the full 8260 list of analytes; the spiking level is 50 ug/L. Check with the project manager for project specific spiking criteria; some projects may require all target analytes are spiked.
- C. Calculate % recovery for all compounds using Calculations equations 1 and 2. All compounds are evaluated to determine acceptance of the MS/MSD. Acceptance criteria are the same as the LCS criteria and can be found in the LIMS.
- D. Whenever the MS and/or MSD recoveries are outside the control limits, check the LCS and LCSD to verify the system was in control, and include this in the case narrative.
- E. Precision is calculated as %RPD. Acceptance criteria are listed in the LIMS. Failures are noted in the narrative.
- F. Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
- G. If recoveries for both the MS/MSD are outside the control

limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.

- H. 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. Additionally, soil weights may vary significantly for some MS/MSD pairs and will affect the RPD calculation.
8. QC and Sample Preparation:
- A. Aqueous sample - place the full 44 mL VOA vial on the SOLATEk autosampler. 5 uL of the internal standard/surrogate standard will be added automatically. All dilutions must be prepared using volumetric flasks or performed by the autosampler. To prepare MB and LCS/LCSD samples use reagent water.
- B. Low-level soil - SOLATEk autosampler is used, place the 5.0 grams of sample in a 44 mL vial and add 5.0 mL of reagent water. Place on the autosampler. The SOLATEk will add 5.0 mL of reagent water and the internal/surrogate standards. To prepare LCS/LCSD samples use blank sand.
- C. Medium/High level soil and Concentrated Waste - medium/high level soil and concentrated waste samples are prepared by weighing 5.0 grams of sample into a 44 mL VOA vial. Add 5.0 mL Methanol. Shake the vial to extract the volatiles into the solvent. A 1000 uL extract aliquot is diluted into a 50mL volumetric flask containing deionized water. The contents are then transferred to a 44mL VOA vial. The SOLATEK autosampler will add the internal standard/surrogate solution to yield a concentration of 50ug/L. To prepare LCS/LCSD samples use reagent water.
- D. EnCore™ sampler (En Chem, Inc)
- D.1 Preservation - A 200g/L sodium bisulfate solution is prepared by weighing 200g of sodium bisulfate and bringing this to volume with deionized water, in a 1L volumetric flask. 5mL 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.
- D.2 Load the vial onto the SOLATEk autosampler. The autosampler will add 5 mL of deionized water containing the internal standard and surrogate standard.
9. Sample Analysis:
- A. Samples may be screened by GCAL.
- A.1 Reasons for screening may include but are not limited to the following:

- A.1.1 Inconsistent historical data
- A.1.2 Physical appearance of the sample
- A.1.3 Client notification of suspected high concentration of VOC's
- A.1.4 No historical data
- A.2 Samples that are deemed necessary for screening must have adequate volume for the screening process and the subsequent volume for the reportable analysis.
- A.3 Screening data is not reportable, as the screening analysis is performed on an instrument that has not met acceptable criteria for reportable analysis. The screening data may be provided upon client request, if applicable.
- A.4 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 VOC's and resulting in instrument down time.
- A.5 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:
 - A.5.1 Client notification of suspected high concentrations of VOC's
 - A.5.2 Physical appearance of the sample
 - A.5.3 Historical data from similar projects
- B. All samples are spiked with the following surrogates: 1,2-Dichloroethane-d4, Toluene-d8, Dibromofluoromethane, and Bromofluorobenzene
 - B.1 All surrogates recoveries must be within the ranges as listed in the LIMS.
 - B.2 If any surrogate is below QC limits and target analytes are present, then the sample must be reanalyzed. If the same surrogate still fails in the same manner, then matrix interference is indicated and is included in the case narrative.
 - B.3 If the recoveries for surrogates are above the upper control limits and no target analytes are detected, the sample is reported with a case narrative indicating surrogate recovery above control limits.
 - B.3 Control limits are updated annually. Project specific control limits and corrective action may apply and may be more stringent. See SOP GEN-019. All project specific limits shall be in the LIMS.
- C. Internal Standards (IS) - All samples are spiked with Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4.
 - C.1 The Internal Standard area for any internal standard must not deviate by more than (-70% to + 100%) from the daily CCV.
 - C.2 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.
 - C.3 If the sample fails any of the IS criteria it must be reanalyzed at the same dilution. If it is apparent

- that the IS is outside criteria due to interference from sample matrix, then contact the client to determine a course of action.
- C.4 Project specific criteria may apply and may be more stringent. DOD and AFCEE projects require the internal standard response drift be evaluated using the most recent ICAL mid-point with control limits of -50 to +200%.
- D. All dilutions are prepared in volumetric flasks or by using the dilution feature on the autosampler. Dilute samples so that the on-column amount is approximately in the mid-range of the calibration curve.
- E. Targets 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:
- E.1 The intensities of the characteristic ion(s) maximize within one scan.
- E.2 The RRT is within ± 0.06 RRT units of the RRT of the standard.
- E.3 The relative intensities of the characteristic ion(s) agree within 30% of the relative intensities of these ions in the reference spectrum.
- E.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.
- F. 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.
- G. TICS may be reported if requested by manually entering data into the LIMS. If no TICS were found, and the client requested a TIC search, this shall be noted in LIMS. A data system library search is used for identification, and the concentration is estimated using an RF of 1 and the area and concentration of the internal standard with the closest retention time.
- H. Manual integrations shall be performed as appropriate. The supervisor reviews manual integrations and the raw data is flagged. For additional information see SOP QA-010.

CALCULATIONS

$$1. \text{ MS } \% \text{ REC} = \frac{\text{MS Concentration-Sample Concentration}}{\text{spike added}} \times 100$$

$$2. \text{MSD } \% \text{ REC} = \frac{\text{MSD Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$3. \% \text{ RPD} = \frac{\text{MS} - \text{MSD}}{\frac{\text{MS} + \text{MSD}}{2}} \times 100$$

$$4. \text{Response Factor} = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak Area of analyte or surrogate

A_{is} = Peak Area of Internal standard

C_s = Concentration of the analyte or surrogate

C_{is} = Concentration of Internal standard

$$5. \% \text{Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

$$6. \text{Concentration using RF:} \\ \text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)}{(A_{is})(\text{RF})(1000)}$$

$$\text{Concentration (mg/kg)} = \frac{(A_s)(C_{is})(D)}{(A_{is})(\text{RF})(W_s)(1000)}$$

A_s = Area of peak for the analyte in sample

D = Dilution factor

RF = Mean Response factor from initial calibration
(area/ng)

A_{is} = Area of internal standard in sample

C_{is} = Concentration of internal standard

W_s = Weight of sample

$$7. \text{Concentration using linear curve fit:} \\ \text{Concentration } (\mu\text{g/L}) = [m(A_s/A_{is}) + b]C_{is}D$$

$$\text{Concentration (mg/kg)} = [m(A_s/A_{is}) + b]C_{is}D(5/W_s)$$

m = Inverse of slope

A_s = Area of peak for the analyte in sample

A_{is} = Area of internal standard in sample

b = Intercept of the y-axis

C_{is} = Concentration of internal standard

D = Dilution factor

W_s = Weight of sample

8. For quadratic curve fit see Target3 Data Analysis Software Reference Guide page 3-17. The quadratic calibration option cannot be used for South Carolina projects.

$$9. \text{Concentration of TICS:} \\ \text{Concentration (mg/kg)} = \frac{(A_s)(C_{is})(D)}{(A_{is})(1000)}$$

A_s = Area of peak for the analyte in sample

D = Dilution factor

A_{is} = Area of internal standard in sample

C_{is} = Concentration of internal standard
 W_s = Weight of sample (if aqueous sample $W_s = 1$ and report in $\mu\text{g/L}$)

10. % Difference = $[(RF_I - RF_C) / RF_I] 100$

RF_I = Average response factor from initial calibration
 RF_C = Response factor from current verification check standard

11. % Drift = $\frac{|\text{Measured Conc} - \text{Spike Conc}|}{\text{Spike Conc}} * 100$

12. % RSD = $(SD / X) 100$

RSD = Relative Standard Deviation
X = mean of 5 initial RF's for a compound
SD = Standard Deviation of average RF's for a compound

REPORTING LIMIT

Samples analyzed by Method 8260B have a routine reporting limit of 5 ppb for all analytes of interest with the exception of the following compounds:

Acetone - 25 ppb
Acetonitrile - 100 ppb
Acrolein - 25 ppb
Acrylonitrile - 25 ppb
1,4-Dioxane - 200 ppb
Isobutanol - 100 ppb
Methyl Ethyl Ketone - 25 ppb
Methyl Methacrylate - 100 ppb
Propionitrile - 100 ppb
Methylene Chloride - 10 ppb
Benzal Chloride - 20 ppb
n-Butyl Alcohol - 100 ppb
Ethyl Acetate - 100 ppb
Ethyl Ether - 100 ppb
Cyclohexanone - 100 ppb
Cyclohexane - 100 ppb
1,3-Butadiene - 10ppb
1,3-Dichloropropene - 10 ppb
1-nitropropane - 10 ppb
2-methyltetrahydrofuran - 20 ppb
2-methyltetrahydropyran - 10 ppb
2-nitropropane - 20
2-H-tetrahydropyran - 20 ppb
Allyl Chloride - 25 ppb
Chloroprene - 80 ppb
Cumene hydroperoxide - 20 ppb
Ethylene oxide - 100 ppb
Tetrahydrofuran - 100 ppb
Sec-Butanol - 10 ppb
t-Butanol - 10 ppb

Lower limits may be achieved for specific projects. Reporting limits are achieved by running a low level standard at the same level as the reporting limit. The RL should be at least 2X the MDL. Specific project criteria may apply. See SOP GEN-019.

METHOD

PERFORMANCE

1. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.
2. This method has been tested using purge-and-trap (Method 5030) in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 ug/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 6 of the 8260 method. Calculated MDLs are present in Table 1 of the 8260 method.
3. The method was tested using purge-and-trap (Method 5030) with water spiked at 0.1 to 0.5 ug/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 7 of the 8260 method. MDL values were also calculated from these data and are presented in Table 2 of the 8260 method.
4. Single laboratory accuracy and precision data were obtained for the Method 5035 analytes in three soil matrices; sand; a soil collected 10 feet below the surface of a hazardous landfill, called C-Horizon; and a surface garden soil. Sample preparation was by Method 5035. Each sample was fortified with the analytes at a concentration of 4 ug/kg. These data are listed in Tables 17, 18, and 19 of the 8260 method. All data were calculated using fluorobenzene as the internal standard added to the soil sample prior to extraction. This causes some of the results to be greater than 100% recovery because the precision of results is sometimes as great as 28%.
5. In general, the recoveries of the analytes from the sand matrix are the highest, the C-Horizon soil results are somewhat less, and the surface garden soil recoveries are the lowest. This is due to the greater adsorptive capacity of the garden soil. This illustrates the necessity of analyzing matrix spike samples to assess the degree of matrix effects.
6. The recoveries of some of the gases, or very volatile compounds such as vinyl chloride, trichlorofluoromethane and 1,1-dichloroethene are somewhat greater than 100%. This is due to the difficulty encountered in fortifying the soil with these compounds, allowing an equilibration period, extracting them with a high degree of precision. Also, the garden soil results in Table 19 of the 8260 method include some extraordinary high recoveries for some aromatic compounds, such as toluene, xylenes, and trimethylbenzenes. This is due to contamination of the soil prior to sample collection, and to the fact that no background was subtracted.

POLLUTION
PREVENTION

See QAPP Section 10.2

WASTE

MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC
GCMS SEMI-VOLATILES
STANDARD OPERATING PROCEDURE

PROCEDURE: GCMSSV-001
PAGE: 1 OF 16
EFFECTIVE DATE: 4/16/07
APPROVED BY: *RLW*
QA/QC APPROVED: *JDT*

SUBJECT

SCOPE AND APPLICATION

EPA Method 8270C (SW-846) 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. BNAs are generally extracted using SOPs EXT-001 for solids and EXT-003 for aqueous samples. High-level waste are prepared by waste dilution. A list of target analytes is available in the LIMS.

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 8270 (SW-846). 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 quadrupole 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.

MATRIX

Water and Solid Extracts

REFERENCE

SW846 8270C and 8000B

HOLDING TIME &
PRESERVATIVE

Waters - store at $4 \pm 2^\circ\text{C}$ for up to but not exceeding seven days before extraction. Hold up to 40 days after extraction at $-15 \pm 5^\circ\text{C}$ in sealed vials. Protect from light.

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Solids - store at $4 \pm 2^\circ\text{C}$ for up to but not exceeding fourteen days before extraction. Hold up to 40 days after extraction at $-15 \pm 5^\circ\text{C}$ in sealed vials. Protect from light.

DEFINITIONS

See SOP GEN-016

SAFETY

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. In all cases consult the applicable material safety data sheet (MSDS) and the supervisor or safety officer. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures, solvents and chemicals must be handled in ventilated hoods.

INTERFERENCES

Interferences due to contamination are monitored by analysis of a method blank performed with each batch. Interferences with the parent (quantitation) ion may occur with co-elution and must be monitored by the analyst.

INSTRUMENTATION & APPARATUS

Agilent/5973-6890N or 5975-6890N GC/MS with autosampler
Target data acquisition system
DB-5MS or RTX Ssil MS columns
Volumetric flasks
Syringes - gas-tight
Pasteur pipets
Disposable glass micropipettes
2 mL autosampler vials with crimp tops
Bottles - amber glass with PTFE-lined screw tops or crimp tops

REAGENTS

All organic solvent shall be of pesticide grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.
Methylene chloride (DCM)
Helium gas

STANDARDS

All standards used must pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

1. All stock standards are received in glass sealed ampoules.
 - A. The following stock standards are used to prepare solutions for calibration and CCV samples:
 - A.1 Stock calibration standard -A custom-made stock standard (TCL-200 ug/ml level) is purchased from NSI. It is stored in an amber vial in the freezer at $-15^\circ \pm 5^\circ\text{C}$, until the documented manufacturer's expiration date. This standard includes the surrogate.

- A.2 Stock calibration standards - 2000 ug/mL -for Appendix IX compounds - commercially prepared stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ until the documented manufacturer's expiration date.
- A.3 Additional standards may be run that are project specific. All standards shall be certified and stored according to the manufacturer's instructions.
- B. Independent stock standards - standard mixes identical to those listed above from a secondary source or a different lot number from the same manufacturer. These standards are used to prepare the ICV.
- C. Stock Internal Standard Mixture: 4000 ug/mL - commercially prepared in methylene chloride, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ until the documented manufacturer's expiration date.
- D. Tuning Standard: 1000 ug/mL - commercially prepared in methylene chloride, stored at $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ until the documented manufacturer's expiration date.
2. Working standards - working standards are prepared by diluting the stock standard as described below.
 - A. Assemble the necessary glassware, syringes and solvent. Spectroscopic grade methylene chloride is used in making all standards.
 - B. 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.
 - C. Fill the volumetric glassware to approximately 75 percent volume.
 - D. Add the proper volume of standards using a syringe.
 - E. After the addition of standard, dilute to the calibration mark in the volumetric flask
 - F. Insert the glass stopper and mix.
 - G. Using a Pasteur pipette, transfer the standard to a 2 mL ampoule. Seal the ampoule immediately.
 - H. Label the vial with the following information:
Expiration Date
Standard ID #
 - I. Store the vial the same as the stock standard.
 - J. Dispose of excess materials in accordance with established laboratory procedures.
 - K. Always quantitate new standards against known standards to

- L. Fill out the standards logbook in accordance with proper laboratory procedure and enter required information in LIMS.
3. The working standards are prepared in the following manner:
- A. Working Appendix IX standards - prepare a 200 ug/mL standard mix from the 2000 ug/mL stock standards.
- B. Working standards from 0.2 ug/mL to 160 ug/mL are prepared as needed before a calibration is performed. They are stored in amber vials in the freezer at $-15^{\circ} \pm 5^{\circ}\text{C}$. To make 1.0 mL of each "working standard" of a calibration, do the following:
- Add 0.001 mL of TCL-200 to 0.999 mL DCM* for a 0.2 ug/mL standard
 - Add 0.005 mL of TCL-200 to 0.995 mL DCM* for a 1 ug/mL standard
 - Add 0.05 mL of TCL-200 to 0.95 mL DCM* for a 10 ug/mL standard
 - Add 0.25 mL of TCL-200 to 0.75 mL DCM* for a 50 ug/mL standard
 - Add 0.40 mL of TCL-200 to 0.60 mL DCM* for an 80 ug/mL standard
 - Add 0.60 mL of TCL-200 to 0.40 mL DCM* for a 120 ug/mL standard
 - Add 0.80 mL of TCL-200 to 0.20 mL DCM* for a 160 ug/mL standard
 - The high concentration standard is the stock standard - 200 ug/mL
 - Add 10 μL of 4000 ng/ μL Internal Standard Mix to each 1.0mL of the "working standard" before shooting

To make the calibration standards for SIM analysis, the TCL standard is used to make the following working and calibration standards.

- Add 125 μL of TCL-200 to 4.875 mL DCM* for a 5 $\mu\text{g}/\text{mL}$ standard
 - Add 200 μL of the 5 $\mu\text{g}/\text{mL}$ standard to 800 μL DCM for a 1 $\mu\text{g}/\text{mL}$ standard.
 - Add 100 μL of the 5 $\mu\text{g}/\text{mL}$ standard to 900 μL DCM for a 0.5 $\mu\text{g}/\text{mL}$ standard.
 - Add 20 μL of the 5 $\mu\text{g}/\text{mL}$ standard to 980 μL DCM for a 0.1 $\mu\text{g}/\text{mL}$ standard.
 - Add 10 μL of the 5 $\mu\text{g}/\text{mL}$ standard to 990 μL DCM for a 0.05 $\mu\text{g}/\text{mL}$ standard.
 - Add 10 μL of 4000 $\mu\text{g}/\text{mL}$ Internal Standard Mix to each 1.0mL of the "working standard" before shooting
- C. The Appendix IX calibration standards are prepared in the same manner from the 200ug/mL working standard. The 200 ug/mL standard will serve as the high concentration standard.

- D. Additional stock standards shall be purchased for compounds not included in the mixes.

GC RECOMMENDED
CONDITIONS

The following lists conditions that may be used in the lab. Conditions vary for each instrument. Adjustments are made when new columns are installed. The analyst is allowed to modify settings to optimize operating conditions.

Initial Temperature: 40°C
Rate A: ~40°C/min to ~245°C
Hold: ~4 to 5 minutes
Injector Temperature: ~ 250°C
Injection volume: 1 uL

PROCEDURE

Analysis Procedure

1. Injection port maintenance is performed every day of use at a minimum. This includes replacement of septa and liner.
2. Tuning - Before analysis begins each GC/MS must meet tuning criteria specified in the method. A solution containing 50 ug/ml each of Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, Pentachlorophenol and Benzidine is analyzed. The DFTPP tuning criteria listed below must be met before proceeding with analysis. DDT breakdown shall not exceed 20% and is calculated as:

$$\% \text{Breakdown} = \frac{\text{area DDE} + \text{area DDD}}{\text{area DDE} + \text{area DDD} + \text{area DDT}} \times 100$$

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 shall be less than 3.0 and for pentachlorophenol shall be less than 5.0. Excess tailing indicates the need for instrument maintenance. If there is no visible tailing document this.

Analyze The DFTPP result using three scans, the apex and the scan prior and following the apex and subtracting the background no more than 20 scans prior to elution of the DFTPP peak. If the DFTPP fails the criteria listed below, then the analyst shall re-tune the instrument, then run another DFTPP. If the DFTPP still fails the criteria, the analyst shall perform instrument maintenance. These criteria must be met once for every 12 hours of analysis time.

DFTPP KEY ION ABUNDANCE CRITERIA

<u>MASS</u>	<u>ION ABUNDANCE CRITERIA</u>
51	30 TO 60% of mass 198
68	less than 2% of mass 69

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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

3. Calibration - The initial calibration consists of a series of standards analyzed at the concentration noted in the Standards Section 3.B-D. An initial calibration curve for each target analyte must be analyzed and evaluated before any peak for that analyte can be quantitated.

The calibration range is defined as the on-column concentration range adjusted for sample prep. 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 (10-200ug/mL):

- At least five contiguous points must be used (six if a quadratic curve fit is employed).
- Calibration acceptance criteria must be met as described below.
- The lowest calibration point must support the lowest reporting limit needed in the associated samples.
- The QC spike amount must be within the calibration range.

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 μ L of 4000 ug/ml Internal Standard Mix to each.

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 8270C. The primary ion is used for quantitation and the secondary ion(s) as confirmation.

The calibration curve is now ready for analysis.

The acceptance criteria for initial calibration must be satisfied before analysis of samples begin. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.

- A.1 Calibration Check Compounds must be less than 30% RSD.
A.2 System Performance Check Compounds must have an (RF) of 0.05 or greater

CCC's

Acenaphthene
Hexachlorobutadiene
Di-n-octylphthalate
Benzo(a)pyrene
2,4-Dichlorophenol
2,4,6-Trichlorophenol
Phenol
Fluoranthene
1,4-Dichlorobenzene
N-Nitroso-di-n-phenylamine
4-Chloro-3-methylphenol
2-Nitrophenol
Pentachlorophenol

SPCC's

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

- B. Additionally one of the following options must be met.

Always attempt to meet calibration criteria using the average response factor. If the average response factor does not pass, options B.2 and B.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 B.4 may be used for some projects if the other options are not successful. The calibration options and requirements are as follows:

B.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 15% RSD for a target analytes, linearity through the origin can be assumed. If the RSD for any analyte is greater than 15%, 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.

B.2 For those compounds that the RSD exceeds 15%, a linear regression equation that is not forced through the origin may be used. The coefficient of determination must be at least 0.995 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.

B.3 A quadratic curve fit may be used if the coefficient of

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.

- B.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:
- The mean of the RSD values for all analytes in the calibration is $\leq 15\%$.
 - Mean RSD = $\frac{\text{Sum of RSD value for each analyte}}{\text{Total \# analytes}}$
 - Mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.
 - Individual compound RSD not to exceed 50%.

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.

- 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.
- 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.

3. INITIAL CALIBRATION VERIFICATION

Immediately following the initial calibration procedure or before sample analysis, the analyst shall perform initial calibration verification (ICV). This will consist of a solution containing all target analytes prepared from a standard that is independent from the initial calibration.

The ICV must be analyzed following the initial calibration. The ICV recovery must be 75-125% except for the compounds listed in Appendix A, which must meet a percent recovery of 50-150%. Specific project criteria may apply that must be achieved for the associated samples. See SOP GEN-019.

If any target analyte recovery is outside the control limits, 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.

4. CONTINUING CALIBRATION VERIFICATION -

If the instrument has been previously calibrated, then a continuing calibration is required. A mid-level standard, 50 ug/mL for scan and 0.5 ug/mL for SIM is analyzed and checked against the initial calibration for verification every 12 hours. CCC's and SPCC's must meet the specified criteria. Calibration Check Compounds must have a %D of ≤ 20 . SPCC's are required to maintain a relative response factor (RF) of 0.05. If there are any deviations, the calibration check standard must be re-analyzed or if necessary, column maintenance should be performed. If the calibration check standard still fails the criteria, then another initial calibration must be run.

All other targets must have a %difference/drift $\leq 30\%$ except for method defined poor performers, listed in Appendix A. Project specific limits may apply that must be achieved for the associated samples. See SOP GEN-019.

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.

5. SAMPLE ANALYSIS

Samples can be analyzed after an acceptable CCV. Add 10 uL of the internal standard mixture to each extract before analysis.

Compound identification - positive hits are achieved by the following criteria:

- A. The quantitation ion used shall be the ion listed in the reference method 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.
- B. The characteristic m/z's must maximize within one scan of each other.
- C. 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 .
- D. The relative abundance of the three characteristic m/z's in the sample must fall within $\pm 30\%$ of the relative abundances of that parameter in the standard.
- E. Structural isomers can be differentiated only if the height between the baseline and the valley between the pairs is less than 25% of the sum of the two peak heights. Otherwise they must be reported as isomeric pairs.
- F. If samples have compounds that are above the calibration range, the sample must be diluted. 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 40 $\mu\text{g/L}$. 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

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.

- G. Manual integrations shall be performed as appropriate. Manual integrations undergo several layers of review and the raw data is flagged. For additional information see SOP QA-010.
- H. 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% and 200% of the internal standard response in the mid-point of the ICAL. Retention time must be within ± 0.5 minutes. If criteria is not met reanalyze at a dilution for the affected analytes or report with a narrative.

QUALITY CONTROL METHOD BLANK

- 1. The method blank (extraction blank) is analyzed with each preparatory batch to demonstrate the extraction procedure did not introduce contamination. Run batch QC in the same analytical batch whenever possible. The batch QC must be analyzed with at least one client sample from the prep batch. This blank also establishes the instrument to be free of contamination.
- 2. No target analytes should be detected in the method blank above one half the reporting limit. If any target analytes are detected, data shall not be reported and samples must be re-extracted and re-analyzed unless the following apply.
 - A. If a target analyte is detected above the reporting limit, data may be reported if the concentration is not greater than 5% of the measured concentration in associated samples. Include a narrative with the data.
 - B. If a target analyte is detected in the method blank but there are no hits in the samples, the data may be reported with a narrative.

SURROGATES

- 1. The surrogates are used to verify that each sample was properly extracted and is not adversely affected by the sample matrix. A surrogate is a non-target compound that is chemically similar to the analytes. The surrogates used are listed in the table below. Surrogates are spiked in each sample at the levels listed in Table 1. Some samples use the same extract for scan and SIM analysis. In these cases the surrogates will be spiked at the scan level.
- 2. Control limits are evaluated after sample analysis. Determine for each sample the project-required limits. If no specific limits are needed, evaluate the recoveries using step 3. If specific limits are needed evaluate using step 4. Evaluate limits as soon as possible to allow necessary repreps to be performed within holding time.

3. Laboratory derived control limits are listed in the LIMS. One surrogate in each fraction (base-neutral or acid) may have a recovery outside the control limits. When this occurs, report the data with a narrative. If more than one surrogate recovery for each fraction is below the lower control limit, re-extract and re-analyze the sample. If the recoveries are outside control limits in the re-extract sample, then indicate in the case narrative and state that the recovery was outside the control limits due to sample matrix. If re-extraction cannot be performed due to insufficient sample, report the data with a narrative. If the surrogate recovery is above the control limits and the sample results are less than the reporting limit, the data may be reported with a narrative.
4. Project specific recovery limits may apply that must be achieved for the associated samples. See SOP GEN-019. Some projects require reprep and/or reanalysis if any surrogate fails recovery criteria either high or low. Check project criteria before proceeding.

Table 1
Surrogates

Surrogates	Scan Conc. mg/L (On column)	SIM Conc mg/L (On column)
Acid Surrogates:		
2-Fluorophenol	100	2
Phenol-d5	100	2
2,4,6-Tribromophenol	100	2
Base - Neutral Surrogates:		
Nitrobenzene-d5	50	1
2-Fluorobiphenyl	50	1
Terphenyl-d14	50	1

LABORATORY CONTROL STANDARD (LCS/LCSD)

1. A LCS and LCSD are included in each preparatory batch to demonstrate the system is in control. Routinely the LCS/LCSD will be spiked with the full list of analytes. The control limits for waters and solids are available in the LIMS. Control limits may be project specific and must be achieved for the associated samples. See SOP GEN-019. Evaluate the data and perform corrective action based on the project limits. Run batch QC in the same analytical batch whenever possible. The batch QC must be analyzed with at least one client sample from the prep batch.
2. If a recovery is above the upper control limit and the sample results are below the reporting limit, the data may be reported with a narrative. Check project criteria. Required corrective action may differ in different projects and must be followed for associated samples.
3. If a recovery is below the lower control limit or precision fails, the entire batch must be re-extracted and re-analyzed.

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If a re-extraction is not possible due to insufficient sample volume, report the data with a narrative.

4. If the full list of target analytes is spiked, a small number of sporadic failures will be allowed. The failure cannot include a primary compound of interest. See Table 2 for the number of allowable failures. The failures are noted in the case narrative. These failures shall be monitored by the QA Department to ensure randomness. This shall be defined as not exceeding failure in 1 of 3 LCS determinations.

Table 2
Number of Allowable Failures

Number of Analytes	Failures Allowed
<11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

MATRIX SPIKES (MS/MSD)

1. The purpose of the MS/MSD is to assess the performance of the method for a particular sample matrix. The recovery criteria for waters and solids are available in the LIMS and are typically the same as the LCS recovery criteria. Project specific recovery and RPD limits may apply, and the data evaluated and corrective action performed based on project requirements.
2. The MS/MSD spike includes the full list of analytes.
3. Whenever the MS and/or MSD recoveries are outside the control limits, check that the LCS and LCSD passed criteria to verify the system was in control. If the LCS and LCSD recoveries and precision are acceptable then continue processing the data. If the LCS and/or LCSD failed then follow the procedure in the LCS section.
4. Review data to verify that a lab error has not occurred (wrong spike amount, not spiked) before automatically identifying a failure as matrix interference.
5. If recoveries for both the MS/MSD are outside the control limits and the recoveries are similar, the data is reportable with a narrative stating the LCS recoveries were acceptable. The failure is attributed to sample matrix.
6. Precision is calculated as %RPD. Acceptance criteria are listed in the LIMS. Failures are noted in the narrative.

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.
8. Spikes may be diluted out in the analysis process. Document this in the case narrative. The spike is diluted out if the dilution is a 10X or greater. MS/MSD are analyzed at the same dilution as the parent sample.

SAMPLE RE-EXTRACTION

Samples are to be re-extracted due to failed QC or due to the sample results. When a MB, LCS, or LCSD fails to meet criteria, the entire batch is sent to extractions for re-extraction. If the MS, MSD, or surrogates fail criteria, only the affected samples are sent to extractions. Particular samples may be re-extracted if the sample results do not match historical values, if a sample and a duplicate do not match, or if physical differences are noted in samples. If samples are re-extracted outside of method specified holding times, both analyses are reported. To request sample/batch re-extraction, do the following:

1. If the extract has been analyzed, process the file and load to LIMS.
2. Enter the code "RP" into the analysis code; this will schedule new sample prep.
3. Complete the Re-extraction Request and Tracking Form (attached) and submit a copy to extraction supervisor.
4. When new extracts are brought to the lab, complete the original Re-extraction Request and Tracking Form and report the data appropriately.
5. If additional sample is not available to re-prepare, check with project manager to determine appropriate action to report the available data.
6. If two sets of data will be reported, see login to obtain a re-extracted sample number.

CALCULATIONS

$$1. \text{ MS \% REC} = \frac{\text{MS Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$2. \text{ MSD \% REC} = \frac{\text{MSD Concentration} - \text{Sample Concentration}}{\text{spike added}} \times 100$$

$$3. \text{ \% RPD} = \frac{\text{MS} - \text{MSD}}{(\text{MS} + \text{MSD}) / 2} \times 100$$

LCS/LCSD results are substituted to calculate %RPD between LCS and LCSD. Results are calculated using the concentration (not percent recovery).

$$4. \text{ Response Factor} = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak Area of analyte or surrogate

A_{is} = Peak Area of Internal standard

C_s = Concentration of the sample or surrogate

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C_{is} = Concentration of Internal standard

$$5. \text{ Surrogate/LCS Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

6. Concentration using RF:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_t)}{(A_{is})(RF)(V_s)}$$

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_t)}{(A_{is})(RF)(W_s)}$$

A_s = Area of peak for the analyte in sample

D = Dilution factor

RF = Mean Response factor from initial calibration

A_{is} = Area of internal standard in sample

C_{is} = Concentration of internal standard in $\mu\text{g/mL}$

W_s = Weight of sample extracted in g

V_t = Total volume of concentrated extract in μL

V_s = Volume of aqueous sample extracted in mL

7. Concentration using linear curve fit:

$$\text{Concentration } (\mu\text{g/L}) = [m(A_s/A_{is}) + b]C_{is}D$$

$$\text{Concentration } (\text{mg/kg}) = [m(A_s/A_{is}) + b]C_{is}D(5/W_s)$$

m = Inverse of slope

A_s = Area of peak for the analyte in sample

A_{is} = Area of internal standard in sample

b = Intercept of the y-axis

C_{is} = Concentration of internal standard in $\mu\text{g/mL}$

D = Dilution factor

W_s = Weight of sample extracted

8. Concentration using a quadratic curve fit:

$$\text{Concentration } (\text{mg/kg}) = [b + a(A_s/A_{is}) + a_2(A_s/A_{is})^2]C_{is}D(5/W_s)$$

9. Concentration of TICS:

$$\text{Concentration } (\text{mg/kg}) = \frac{(A_s)(C_{is})(D)}{(A_{is})(1000)}$$

A_s = Area of peak for the analyte in sample

D = Dilution factor

A_{is} = Area of internal standard in sample

C_{is} = Concentration of internal standard

W_s = Weight of sample (if aqueous sample $W_s = 1$ and report in $\mu\text{g/L}$)

10. % Difference = $[(RF_I - RF_C) / RF_I] \times 100$ where:

RF_I = Average response factor from initial calibration

RF_C = Response factor from current verification check standard

11. % Drift = $\frac{(\text{Calculated Conc.} - \text{Theoretical Conc.})}{\text{Theoretical Conc.}} \times 100$

12. % RSD = $(SD / X) \times 100$ Where:

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X = mean of 5 initial RF's for a compound
SD = Standard Deviation of average RF's for a compound

REPORTING LIMITS Quantitation limits (PQLs) are defined by the low-level calibration standard. Default reporting limits are employed for reporting and cannot be lower than the PQL. The PQL cannot be lower than the estimated MDL and project specific requirements may define the relationship between the PQL and the MDL that must be achieved for the associated samples. See SOP GEN-019.

METHOD
PERFORMANCE

Method 8250 (the packed column version of Method 8270) was tested by 15 laboratories using organic-free reagent water, drinking water, surface water, and industrial wastewater spiked at six concentrations ranging from 5 to 1,300 ug/L. Single operator accuracy and precision, and method accuracy were found to be directly related to the concentration of the analyte and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7 of Method 8270C. These values are presented as guidance only and are not intended as absolute acceptance criteria.

Internal control limits are generated for laboratory QC samples including LCS/LCSD, MS/MSD, and surrogates.

POLLUTION
PREVENTION

See QAPP Section 10.2

WASTE
MANAGEMENT

See SOP GEN-009

APPENDIX A POOR PERFORMING COMPOUNDS

Benzidine	1,4-Naphthoquinone	4-Nitroquinoline-1-oxide
Benzoic Acid	m-Dinitrobenzene	Parathion
2-Picoline	Pentachlorobenzene	Metapyrilone
n-Nitrosomethylethylamine	2-Naphthylamine	Isodrin
Methyl methanesulfonate	2,3,4,6-Tetrachlorophenol	Aramite
n-Nitrosodiethylamine	1-Naphthylamine	p-(Dimethylamino) azobenzene
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		

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STANDARD OPERATING PROCEDURE
REVIEW FORM

GCAL
Standard Operating Procedure for MSSV 8270

Procedure: GCMSSV-001
Revision: 14
Effective Date: 4/16/07

Reviewed By/Date: RLW 08-25-08

Approved By/Date: JDT 8/26/08

No changes needed.

GULF COAST ANALYTICAL LABORATORIES
EXTRACTIONS
STANDARD OPERATING PROCEDURES

PROCEDURE: EXT-002
PAGE: 1 of 5
EFFECTIVE DATE: 10/07/2008
APPROVED BY: MAP
QA/QC APPROVED: JDT

SUBJECT SCOPE AND APPLICATION

A 30 gram portion of organic fraction is mixed with anhydrous sodium sulfate and extracted with methylene chloride/acetone using an ultrasonic probe. If peaks are present at greater than 10,000 ug/kg, discard the extract and prepare the sample by the medium level method.

MATRIX Solid

REFERENCE SW846 Method 3550C

PRESERVATIVE Cool 4°C

HOLDING TIME Samples extracted within 14 days and extracts analyzed within 40 days following extraction

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

APPARATUS 250 mL beaker
Kuderna Danish (K-D) apparatus
a) Concentrator tube, 10mL graduated
b) Evaporation flask, 500mL (attach to tube with blue clamp)
c) Synder column
Sonicator, Fisher Scientific Ultrasonic Dismembrator equipped with ¾ inch disruptor horn. Misonex sonicators are also available as a backup.
Filter paper - FisherBrand Q8, 125mm
Analytical balance
0.5 mL syringe
1.0 mL syringe

1.0 mL Class A volumetric flask
 5.0 mL Class A volumetric flask
 10.0 mL Class A volumetric flask

REAGENT All organic solvent shall be of pesticide grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.
 1:1 Methylene chloride/Acetone
 Methylene chloride
 Acetone
 Hexane
 Sodium sulfate - granular, anhydrous - heat at 400°C for a minimum of 4 hours. Store prepared sodium sulfate in glass screw cap jars. Keep covered at all times when not in use. Record preparation in logbook.

STANDARDS Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

1. Surrogate Standard Spiking Solution

Surrogate standards are added to all samples (including QC). The surrogates for this procedure are Tetrachloro-m-xylene and Decachlorobiphenyl. For method 8082, only Decachlorobiphenyl is required although both surrogates are included in the standard.

Stock surrogate standard - 200 ug/mL Tetrachloro-m-xylene and Decachlorobiphenyl.

Working surrogate standard - 0.5 ug/mL - Prepare by adding 0.5 mL of the stock surrogate standard to a 200 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in teflon-sealed containers. The standards must be replaced after six months, when stock standard expires, or sooner if comparisons with quality control check samples indicate a problem.

2. Pesticide Spiking Solutions

Stock 608 spike - 1000 ug/mL of:

Aldrin	Endosulfan sulfate
alpha-BHC	Endrin
beta-BHC	Endrin aldehyde
delta-BHC	Endrin ketone
gamma-BHC	Heptachlor
4,4'-DDD	Heptachlor epoxide
4,4'-DDE	Methoxychlor
	Endosufan I
Dieldrin	alpha-chlordane
Endosulfan II	gamma-chlordane

Working 608 spike - 1 ug/mL - Prepare by adding 250

uL of the stock standard to a 250 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in teflon-sealed containers. The standards must be replaced after six months, when stock standard expires, or sooner if comparisons with quality control check samples indicate a problem.

3. PCB Spiking Solutions (8082)

Stock standard - 5000 ug/mL individual standards of Aroclor-1260 and Aroclor-1016

Working PCB spike - 4 ug/mL - Prepare by adding 400 uL of the stock standards to a 500 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in teflon-sealed containers. The standards must be replaced after six months, or sooner if comparison with quality control check samples indicate a problem.

Projects may require spiking with an Aroclor other than 1016/1260. Stock standards will be ordered as needed in this case. Working spikes will be prepared at 4 ug/mL.

PROCEDURE

Sample Extraction

1. Label each beaker with the sample ID of the sample or QC sample to be extracted. Complete the extraction sheet with the sample ID's.
2. For the method blank, LCS and LCSD, weigh 30 grams sodium sulfate into a 250 mL beaker. Spike the blank, LCS and LCSD with 1.0 mL of the working surrogate standard. For method 8081 add 0.5 mL of the working 608 spike to the LCS and LCSD. For method 8082, add 1 mL of the PCB working spike to the LCS and LCSD.
3. Homogenize sample by pouring the contents of the sample onto a piece of butcher paper, chop and mix with a tongue depressor or spatula. Remove any foreign objects such as sticks, leaves, or rocks. Weigh approximately 30g of homogenized sample to the nearest 0.1g into a 250mL beaker and add anhydrous sodium sulfate. Mix well. The sample should have a sandy texture at this point. Add 1.0 mL of the surrogate working standard to each sample and the spiked samples. For method 8081A, add 0.5 mL of the working 608 spike to the MS and MSD. For method 8082, add 1 mL of the PCB working spike to the MS and MSD.
4. Immediately, add at least 100 mL of 1:1 Methylene Chloride/Acetone to each beaker. The solvent

level should be about 2 inches above the sample in the beaker.

5. Sonicate for three minutes with the W-385 (or three minutes with the W-375), using a N. 2083/4 inch standard disrupter horn with output control knob set at 6 (or No. 3053/4 inch tapped high gain "Q" disrupter horn set at 5) and mode switch on "1 second pulse" and percent duty cycle knob set at 50 percent. Do NOT use MICROTIP probe.
6. Decant and filter extracts through FisherBrand Q8 glass filters into a K-D concentrator consisting of 10 ml concentrator tube and a 500 ml flask. The flask contains 1 boiling chip.
7. Repeat extraction two more times with two additional 100 mL portions of 1:1 Methylene chloride/Acetone. Decant off the extraction solvent after each sonication. On the final sonication, filter the entire sample and rinse filter with Methylene chloride.
8. Attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1.0 mL of Methylene chloride to the top. Place the K-D apparatus on a steam generator (100°C). 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 the extract reaches 10mL, add 50 mL of pesticide-grade hexane through the Snyder column. Concentrate the solvent extract as before. The elapsed time of concentration should be about 5-10 minutes. When the apparent volume of liquid reaches 1.0 mL, remove the K-D apparatus and cool and drain for 10 minutes as above.
9. Remove the Snyder column, wipe the joint between the flask and concentrator tube with a Kim-wipe, in order to remove excess water, and rinse the flask and its lower joint with 1.0 to 2.0 mL of hexane from a Teflon wash bottle. Adjust the final extract volume to 10.0 mL using a 10.0 mL Class A volumetric flask. NOTE: For adjusted RDL's use appropriate 1.0 mL or 5.0 mL Class A volumetric flasks.
10. If the analyst determines that clean-up is required, remove a 1.0 ml aliquot of the final extract to perform any required clean-up procedures. Refer to Standard Operating Procedure EXT-058 and EXT-059 for information on Acid/Permanganate clean-up and Florosil clean-up.

11. Record the standard ID's and solvent Lot numbers on the extraction sheet. Enter the sample prep information in the LIMS. Deliver the extracts and a copy of the extraction sheet to the GC-SV Laboratory. If no one is available to receive the extracts, place the extracts in the appropriate refrigerator.

12. Samples are analyzed by SOP GC-013 and GC-023

METHOD
PERFORMANCE

- 1) The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
- 2) The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

METHOD
MODIFICATION

Procedure step 4 - The extraction solvent is added to the sample to be approximately 2 inches above the solid sample in the beaker. Exactly 100mL is not measured as stated in the method.

GULF COAST ANALYTICAL LABORATORIES
EXTRACTIONS
STANDARD OPERATING PROCEDURES

PROCEDURE: EXT-002
PAGE: 1 of 5
EFFECTIVE DATE: 10/07/2008
APPROVED BY: MAP
QA/QC APPROVED: JDT

SUBJECT SCOPE AND APPLICATION

A 30 gram portion of organic fraction is mixed with anhydrous sodium sulfate and extracted with methylene chloride/acetone using an ultrasonic probe. If peaks are present at greater than 10,000 ug/kg, discard the extract and prepare the sample by the medium level method.

MATRIX Solid

REFERENCE SW846 Method 3550C

PRESERVATIVE Cool 4°C

HOLDING TIME Samples extracted within 14 days and extracts analyzed within 40 days following extraction

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

APPARATUS 250 mL beaker
Kuderna Danish (K-D) apparatus
a) Concentrator tube, 10mL graduated
b) Evaporation flask, 500mL (attach to tube with blue clamp)
c) Synder column
Sonicator, Fisher Scientific Ultrasonic Dismembrator equipped with ¾ inch disruptor horn. Misonex sonicators are also available as a backup.
Filter paper - FisherBrand Q8, 125mm
Analytical balance
0.5 mL syringe
1.0 mL syringe

uL of the stock standard to a 250 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in teflon-sealed containers. The standards must be replaced after six months, when stock standard expires, or sooner if comparisons with quality control check samples indicate a problem.

3. PCB Spiking Solutions (8082)

Stock standard - 5000 ug/mL individual standards of Aroclor-1260 and Aroclor-1016

Working PCB spike - 4 ug/mL - Prepare by adding 400 uL of the stock standards to a 500 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in teflon-sealed containers. The standards must be replaced after six months, or sooner if comparison with quality control check samples indicate a problem.

Projects may require spiking with an Aroclor other than 1016/1260. Stock standards will be ordered as needed in this case. Working spikes will be prepared at 4 ug/mL.

PROCEDURE

Sample Extraction

1. Label each beaker with the sample ID of the sample or QC sample to be extracted. Complete the extraction sheet with the sample ID's.
2. For the method blank, LCS and LCSD, weigh 30 grams sodium sulfate into a 250 mL beaker. Spike the blank, LCS and LCSD with 1.0 mL of the working surrogate standard. For method 8081 add 0.5 mL of the working 608 spike to the LCS and LCSD. For method 8082, add 1 mL of the PCB working spike to the LCS and LCSD.
3. Homogenize sample by pouring the contents of the sample onto a piece of butcher paper, chop and mix with a tongue depressor or spatula. Remove any foreign objects such as sticks, leaves, or rocks. Weigh approximately 30g of homogenized sample to the nearest 0.1g into a 250mL beaker and add anhydrous sodium sulfate. Mix well. The sample should have a sandy texture at this point. Add 1.0 mL of the surrogate working standard to each sample and the spiked samples. For method 8081A, add 0.5 mL of the working 608 spike to the MS and MSD. For method 8082, add 1 mL of the PCB working spike to the MS and MSD.
4. Immediately, add at least 100 mL of 1:1 Methylene Chloride/Acetone to each beaker. The solvent

level should be about 2 inches above the sample in the beaker.

5. Sonicate for three minutes with the W-385 (or three minutes with the W-375), using a N. 2083/4 inch standard disrupter horn with output control knob set at 6 (or No. 3053/4 inch tapped high gain "Q" disrupter horn set at 5) and mode switch on "1 second pulse" and percent duty cycle knob set at 50 percent. Do NOT use MICROTIP probe.
6. Decant and filter extracts through FisherBrand Q8 glass filters into a K-D concentrator consisting of 10 ml concentrator tube and a 500 ml flask. The flask contains 1 boiling chip.
7. Repeat extraction two more times with two additional 100 mL portions of 1:1 Methylene chloride/Acetone. Decant off the extraction solvent after each sonication. On the final sonication, filter the entire sample and rinse filter with Methylene chloride.
8. Attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1.0 mL of Methylene chloride to the top. Place the K-D apparatus on a steam generator (100°C). 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 the extract reaches 10mL, add 50 mL of pesticide-grade hexane through the Snyder column. Concentrate the solvent extract as before. The elapsed time of concentration should be about 5-10 minutes. When the apparent volume of liquid reaches 1.0 mL, remove the K-D apparatus and cool and drain for 10 minutes as above.
9. Remove the Snyder column, wipe the joint between the flask and concentrator tube with a Kim-wipe, in order to remove excess water, and rinse the flask and its lower joint with 1.0 to 2.0 mL of hexane from a Teflon wash bottle. Adjust the final extract volume to 10.0 mL using a 10.0 mL Class A volumetric flask. NOTE: For adjusted RDL's use appropriate 1.0 mL or 5.0 mL Class A volumetric flasks.
10. If the analyst determines that clean-up is required, remove a 1.0 ml aliquot of the final extract to perform any required clean-up procedures. Refer to Standard Operating Procedure EXT-058 and EXT-059 for information on Acid/Permanganate clean-up and Florosil clean-up.

11. Record the standard ID's and solvent Lot numbers on the extraction sheet. Enter the sample prep information in the LIMS. Deliver the extracts and a copy of the extraction sheet to the GC-SV Laboratory. If no one is available to receive the extracts, place the extracts in the appropriate refrigerator.
12. Samples are analyzed by SOP GC-013 and GC-023

METHOD
PERFORMANCE

- 1) The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
- 2) The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

METHOD
MODIFICATION

Procedure step 4 - The extraction solvent is added to the sample to be approximately 2 inches above the solid sample in the beaker. Exactly 100mL is not measured as stated in the method.

GULF COAST ANALYTICAL LABORATORIES
EXTRACTIONS
STANDARD OPERATING PROCEDUREPROCEDURE: EXT-010
PAGE: 1 OF 5
EFFECTIVE DATE: 10/07/2008
APPROVED BY: *MAR*
QA/QC APPROVED: *JOT*

SUBJECT SCOPE AND APPLICATION

This Standard Operating Procedure describes the steps for separating Pesticide/PCB compounds in aqueous samples. A measured volume of sample, 1.0 L at a pH between 5 and 9 is serially extracted with Methylene chloride using a separatory funnel. The extract is dried, concentrated and as necessary exchanged into a solvent compatible with the cleanup or determinative step to be used.

MATRIX Water

REFERENCE EPA Method 608
SW846 Method 3510C

PRESERVATIVE None

HOLDING TIME 7 days

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

INTERFERENCES

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.
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.
3. Glassware contamination resulting in analyte degradation. Certain analytes (Aldrin, Heptachlor and most organophosphorus pesticides) may degrade due to soap residue on glassware. Glassware that is particularly difficult to rinse such as a K-D flask should be hand rinsed very carefully to avoid this problem.

APPARATUS 2000 ml Teflon separatory funnel with Teflon stopcock
Kuderna-Danish (K-D) apparatus*Uncontrolled Copy - For Reference Only*

- A) Concentrator tube, 10 ml, 25 ml, graduated
- B) Evaporation flask, 500 ml (attach to tube with blue clamp)
- C) Snyder column, three-ball macro Boiling chips, Teflon
- Water bath
- Graduated cylinder -- 1 liter
- Volumetric flasks - 200 mL, 250 mL
- pH indicator paper with range between pH 2-14
- 250 and 500 uL syringes
- 1.0 mL syringe
- 1.0 mL Class A volumetric flask
- 5.0 mL Class A volumetric flask
- 10.0 mL Class A volumetric flask

REAGENTS

All inorganic reagents must be reagent grade or equivalent. All organic solvents must be pesticide grade or equivalent. Label all reagents as required by SOP GEN-006.

Deionized water
Sodium sulfate -- granular, anhydrous
Methylene chloride
Hexane
Methanol
5N sodium hydroxide
1:1 sulfuric acid

STANDARDS

1. Surrogate Standard Spiking Solution

Surrogate standards are added to all samples (including QC). The surrogates for this procedure are Tetrachloro-m-xylene and Decachlorobiphenyl. For method 8082, only Decachlorobiphenyl is required although both surrogates are included in the standard.

Stock surrogate standard - 200 ug/mL Tetrachloro-m-xylene and Decachlorobiphenyl.

Working surrogate standard - 0.5 ug/mL - Prepare by adding 0.5 mL of the stock surrogate standard to a 200 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in Teflon-sealed containers. The standards must be replaced after six months, or sooner if comparisons with quality control check samples indicate a problem.

2. Pesticide Spiking Solutions

Stock 608 spike - 1000 ug/mL of:

Aldrin	Endosulfan sulfate
alpha-BHC	Endrin
beta-BHC	Endrin aldehyde
delta-BHC	Endrin ketone
gamma-BHC	Heptachlor
4,4'-DDD	Heptachlor epoxide
4,4'-DDE	Methoxychlor
Endosufan I	alpha-chlordane

Dieldrin
Endosulfan II

gamma-chlordane

Working 608 spike - 1 ug/mL - Prepare by adding 250 uL of the stock standard to a 250 mL volumetric flask and diluting to volume with Acetone. Store the standard at 4°C in Teflon-sealed containers. The standards must be replaced after six months, or sooner if comparisons with quality control check samples indicate a problem.

3. PCB Spiking Solutions (8082)

Stock standard - 5000 ug/mL individual standards of Aroclor-1260 and Aroclor-1016

Working PCB spike - 4 ug/mL - Prepare by adding 400 uL of the stock standards to a 500 mL volumetric flask and diluting to volume with methanol. Store the standard at 4°C in Teflon-sealed containers. The standards must be replaced after six months, or sooner if comparisons with quality control check samples indicate a problem.

Projects may require spiking with an Aroclor other than 1016/1260. Stock standards will be ordered as needed in this case. Working spikes will be prepared at 4 ug/mL.

PROCEDURE

1. Rinse all separatory funnels with Methylene chloride two times. Discard solvent rinse into waste container.
2. Label each separatory funnel with the sample ID to be extracted. Complete the laboratory extraction sheet with the sample IDs.
3. With each extraction batch, a method blank, LCS/LCSD and MS/MSD are performed. A LCSD/MSD are not required for method 608.
4. Shake and mix sample well. Using a 1 L graduated cylinder, measure out a 1 L sample aliquot and place it into a 2 L separatory funnel. If sample size does not permit a full 1 L volume, the volume actually extracted must be recorded in the extraction log and the analysis must reflect the true original volume of sample on the appropriate bench sheet.) In the extraction for TCLP, measure 100 mL of sample. Use 1000 mL of deionized water for the method blank and LCS/LCSD.
5. Check pH of the sample with pH paper by removing a drop of sample with a disposable Pasteur pipet and adjust to between pH 5 and 9 with a **5N Sodium Hydroxide solution** and/or 1:1 sulfuric acid solution.
6. Add 1.0 ml of surrogate standard spiking solution into each sample using a 1 mL syringe.
7. For method 608, add 0.5mL of the working 608 spike to the

LCS and MS. For method 8082, add 1 mL of the PCB working spike to the LCS, LCSD, MS, and MSD.

8. Use 60 ml of Methylene chloride to rinse the sample bottle (if entire contents are used) and graduated cylinder (after rinsing the bottle, transfer to the graduated cylinder). (Note: If significant amounts of sediment are in the bottom of the sample container, it may not be possible to rinse with the solvent). Transfer this rinse solvent to the separatory funnel and extract the sample by shaking the funnel for a minimum of two minutes, with periodic venting to remove excess pressure. 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 glass funnel and collect in a K-D concentrator flask, fitted with a 10 ml graduated receiver. Using the Methylene chloride rinse bottle, rinse the funnel with a stream of Methylene chloride and collect in the K-D concentrator.
9. Add a second 60 ml volume of Methylene chloride to the sample in the separatory funnel and repeat the extraction procedure, combining the extracts in the K-D flask. Perform a third extraction in the same manner. (Note: It is imperative that the K-D flask as well as the separatory funnel be labeled with the sample identification number.) Rinse the sodium sulfate drying tube with 20 to 30 ml of Methylene chloride to complete quantitative transfer to K-D flask.
10. Add one or two clean silicon carbide boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1.0 ml of Methylene chloride to the top. Place the K-D apparatus in a water bath. 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 the extract reaches 10 ml, add 50 ml of pesticide-grade hexane through the Snyder column. Concentrate the solvent extract as before. The elapsed time of concentration should be about 5-10 minutes. When the apparent volume of liquid reaches 1.0 ml, remove the K-D apparatus and cool and drain for 10 minutes as above.
11. Remove the Snyder column, wipe the joint between the flask and concentrator tube with a Kim-wipe in order to remove excess water, and rinse the flask and its lower joint with 1.0 to 2.0 ml of hexane from a Teflon wash bottle. Adjust the final extract volume to 10.0 ml in a 10 mL volumetric flask with Hexane then put in a 2.0 mL vial.

12. Enter the prep batch information in the LIMS. Deliver a copy of the extraction log and the extracts to the GC-Semivolatiles lab. If no one is available to receive the extracts, place the extracts in the appropriate refrigerator.
13. Samples are analyzed by SOP GC-013 and GC-023.

METHOD
PERFORMANCE

- 1) The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
- 2) The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION
PREVENTION

See QAPP Section 10.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
EXTRACTIONS
STANDARD OPERATING PROCEDURE

PROCEDURES: EXT-032
PAGE: 1 OF 3
EFFECTIVE DATE: 12/06/2006
APPROVED BY: *SAB*
QA/QC APPROVED: *AM*

SUBJECT SCOPE AND APPLICATION

The pH of solid or waste samples is determined electrometrically using a combination electrode. The meter is calibrated using a series of standards of known pH.

MATRIX Solid

REFERENCES SW846-9045

PRESERVATIVE None

HOLDING TIME Analyze as soon as possible.

DEFINITIONS See SOP GEN-016

SAFETY Every 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

REAGENTS Deionized water
Buffers 1.00, 4.00, 5.00, 7.00, 8.00, 10.00, 13.00

APPARATUS ORION SA720 pH meter with automatic temperature compensation
Combination Electrode
Temperature Probe
Plastic Specimen Cups
Plastic Cups
Top Loader Balance
Shaker
Spatulas

PROCEDURE CALIBRATION

1. Press [calibrate] button.
2. Meter will ask, how many buffers? Press [3] [yes].
3. Place probe in Buffer (pH 4.00); allow meter to stabilize; press [4.00] [yes].
4. Place probe in Buffer (pH 7.00); allow meter to stabilize; press [7.00] [yes].

5. Place probe in Buffer (pH 10.00); allow meter to stabilize; press [10.00] [yes].
6. Record slope percent in calibration logbook. It will be displayed after 3rd buffer. The slope must be 92-108 for an acceptable calibration. If outside these limits, clean probe and recalibrate.
7. Analyze the QC check buffer. Record result in the logbook. Leave probe in the mid-range buffer.
8. Follow the same procedure to calibrate with a different range of buffers.

SAMPLE PREPARATION AND ANALYSIS

1. Homogenize sample by pouring the contents of the container onto a piece of butcher paper, chop and mix with a tongue depressor or spatula. Remove any foreign object such as sticks, leaves, or rocks. Weigh 20g +/- 0.1g of the homogenized sample into a plastic specimen cup.
2. Add 20 ml of deionized water to sample, cover and shake for 5 minutes on the shaker. Additional water may be added to hydroscopic materials or other problematic matrices. If the waste is hydroscopic and adsorbs all the deionized water, add an additional 80mL of DI water and shake for 5 minutes.
3. Allow sample to sit for about 60 minutes to let the particulates settle. If the solution is multiphasic, decant the oily phase and measure the pH of the aqueous phase.
4. Immerse the electrode and temperature probe in the solution. Adjust the electrode in the holder so that, upon lowering, the electrode and temperature probe will be immersed just below the solution or decant the solution into a plastic cup and immerse the probes in the solution.
5. Press "measure". Allow the reading to stabilize. The display will read ready when the reading is stable. Record the sample number and the result to two decimal places in the logbook. Report three significant figures when reporting data in the LIMS.
6. If a pH result is outside the current calibration range, the meter must be recalibrated with buffers that bracket the pH of the sample.
7. Rinse the probe with deionized water. Repeat steps 1-5 for each sample.

DETECTION LIMIT Lower 2.0
Upper 13.0

QUALITY CONTROL 1. Analyze a mid-range buffer as a QC check after calibration. The determined pH must be within +/- 0.05 pH units. If the QC is out of control limits the meter must

	be re-calibrated.
	2. Duplicate one of every 20 samples.
MAINTENANCE	1. Flush and refill electrode as needed.
	2. When dirt or oil builds up on the electrode, clean with methanol.
METHOD PERFORMANCE	No data provided.
POLLUTION PREVENTION	See QAPP Section 10.2
WASTE MANAGEMENT	See SOP GEN-009

pH LOGBOOK

QC Limits: True Value \pm 0.05 pH units
 Slope Limits: 92-108

Analyst/Date: _____

	Calibration Time	Buffer	Lot #	Expiration Date	Calibration Levels			Slope	QC Check	
					1	2	3		Actual	Found
1										
2										
3										
4										
5										
6										

pH RESULTS

HBN #: _____

	Client	Sample ID	Time	Analyst	Sample wt (g)	ml DI	Result
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

QC Check Lot#: _____ Exp. _____

QC Check Lot#: _____ Exp. _____

STANDARD OPERATING PROCEDURE
REVIEW FORM

GCAL Standard Operating Procedure for <i>PH in solid air</i> <i>WASTE samples</i>	Procedure: <i>EXT-032</i> Revision: <i>8</i> Effective Date: <i>12/06/06</i>
<i>REVISION 8</i>	
Reviewed By/Date: <i>DT 8-25-08</i>	Approved By/Date: <i>JDT 8/25/08</i>

NO CHANGE

GULF COAST ANALYTICAL LABORATORIES, INC.
EXTRACTIONS
STANDARD OPERATING PROCEDUREPROCEDURES: EXT-033
PAGE: 1 of 3
EFFECTIVE DATE: 10/26/2001
APPROVED BY: *SAB*
QA/QC APPROVED: *pbm*

SUBJECT SCOPE AND APPLICATION

The pH of water samples is determined electrometrically using a combination electrode. The meter is calibrated using a series of standard solutions of known pH.

MATRIX Water

REFERENCES EPA 150.1
Standard methods 4500-H⁺B Electrometric method
SW 846 9040B

PRESERVATIVE None

HOLDING TIME Analyze Immediately - Analysis should be performed as soon as possible after receipt

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

REAGENTS Deionized Water (DI)
Calibration Buffers
Buffer 1.0, 4.0, 5.0, 7.0, 8.0, 10.0, 13.0
Each aliquot of the buffers may only be used once

APPARATUS Orion 720A pH meter
Combination electrode
Temperature probe
Magnetic stir plate
Magnetic stirring bars
Plastic cups

PROCEDURE CALIBRATION

1. Press [calibrate] button.
2. Meter will ask, how many buffers? Press [3] [yes]. Each buffer should be poured into a plastic cup containing a magnetic stir bar.
3. Place electrode and temperature probe in the buffer (pH 4.00); stir gently and allow meter to stabilize; press [4.00]

- [yes].
- Place electrode and temperature probe in the buffer (pH 7.00); stir gently and allow meter to stabilize; press [7.00] [yes].
 - Place electrode and temperature probe in the buffer (pH 10.00); allow meter to stabilize; press [10.00] [yes].
 - Record slope percent in calibration logbook. It will be displayed after the 3rd buffer.
 - Analyze the QC check buffer (mid range buffer) immediately after calibration. Pour the QC check buffer into a plastic cup containing a stir bar. Place the electrode and temperature probe in the solution, stir gently and allow to stabilize. The reading must be within 0.05 pH units of the true value. If not, the meter must be recalibrated. Record result in the logbook. A QC sample must be analyzed with every 20 samples.
 - Follow the same procedure to calibrate with a different range of buffers.
 - When the probe is not in use, leave soaking in a pH 7 buffer solution.

SAMPLE ANALYSIS

- Shake samples thoroughly, pour approximately 20 ml into a plastic cup containing a magnetic stir bar. Sufficient sample must be available to cover the sensing element of the electrode and to give adequate clearance for the stir bar.
- Immerse the electrode and temperature probe in the sample. Gently stir at a constant rate to provide homogeneity and suspension of solids. Allow to stabilize.
- Read results to the second decimal place. Post results after rounding to one decimal place.
- Repeat steps 1-3 on successive aliquots of each sample until values differ by less than 0.1 pH units. Two or three aliquots of each sample should be sufficient.
- Thoroughly rinse electrode and temperature probe with DI water.
- Repeat steps 1-5 for each sample. Record the first sample as the batch duplicate for each day.
- When a sample does not fall within the current calibration range, the meter must be re-calibrated in a range to bracket the sample(s). The meter will only store one slope at a time, therefore only the last calibration is valid. Multiple calibrations may be necessary each day.

DETECTION LIMIT Lower 2.00
Upper 13.0

MAINTENANCE 1. Flush and refill electrode as needed.
2. When dirt or oil builds up on the electrode, clean with methanol.

METHOD
PERFORMANCE Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as: Bias %	Accuracy as: Bias pH Units
3.5	0.10	-0.29	-0.01
3.5	0.11	-0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	-0.03	-0.002
8.0	0.13	-0.12	-0.01
8.0	0.12	+0.16	+0.01

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GCAL

GULF COAST ANALYTICAL LABORATORIES, INC.

pH LOGBOOK

QC Limits: True Value \pm 0.05 pH units
Slope Limits: 92-108

Analyst/Date: _____

Calibration Time	Buffer	Lot #	Expiration Date	Calibration Levels			Slope	QC Check	
				1	2	3		Actual	Found
1									
2									
3									
4									
5									
6									

pH RESULTS

HBN #: _____

Client	Sample ID	Time	Analyst	Result 1	Result 2	Result 3
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

Result 1 - Aliquot 1 (Repeat with successive aliquots of each sample until results agree within 0.1 pH units)

Result 2 - Aliquot 2

Result 3 - Aliquot 3

QC Check Lot# _____

Exp. _____

QC Check Lot# _____

Exp. _____

Revision 0, 04/11/07

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STANDARD OPERATING PROCEDURES FOR
REACTIVE CYANIDE AND REACTIVE SULFIDE

REVISION NO. 7

GULF COAST ANALYTICAL LABORATORIES
EXTRACTIONS
STANDARD OPERATING PROCEDURE

PROCEDURE: WL-054
PAGE: 1 OF 4
EFFECTIVE DATE: 02/18/09
APPROVED BY: *MAP*
QA/QC APPROVED: *JDT*

SUBJECT	SCOPE AND APPLICATION
	This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures. This method provides a way to determine the specific rate of release of hydrocyanic acid and hydrogen sulfide upon contact with an aqueous acid. This test measures only the hydrocyanic acid and hydrogen sulfide evolved at the test conditions.
MATRIX	Water and Solid
REFERENCE	SW846 7.3.3.2 and 7.3.4.2
PRESERVATIVE	None - Samples should be stored at $\leq 6^{\circ}\text{C}$
SAMPLE COLLECTION	Samples should be collected with a minimum of aeration. The sample bottle should be completely filled, minimizing headspace.
SAMPLE CHAIN OF CUSTODY	Wet lab personnel scan the barcode of the sample out for analysis in the LIMS to transfer internal custody of the sample. When the analysis is complete, Login personnel will scan the samples back to the cooler to complete the internal chain-of-custody. See QAPP Chapter 8 for more details.
HOLDING TIME	Commence preparation and analysis as soon as possible
DEFINITIONS	See SOP GEN-016
SAFETY	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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.
APPARATUS	Round-bottom flask, 500 mL, two-neck Gas Scrubber - 100 mL Stirring plate Stirring bars Addition funnel

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Flexible tubing
Flowmeter for monitoring nitrogen flow rate
Analytical Balance
Pipets
Mechanical pipet
Graduated cylinders
Wood tongue depressors
Scoopula spatulas
Large carboy
Volumetric flasks
Nitrogen cylinder and regulator
pH paper

REAGENTS &
STANDARDS

Deionized water (DI)
Sulfuric acid - concentrated
Sulfuric acid - 0.01N H_2SO_4 - Add 10.5 mL concentrated H_2SO_4 to the carboy and dilute to 10 gallons with deionized water
Cyanide standard - 1000 mg/L, commercially prepared
Sulfide reference solution - 1000 mg/L hydrogen sulfide - Dissolve 7.50 g of $Na_2S \cdot 9H_2O$ in 1.0 L of deionized water (The true value will be determined by the titrimetric method)
50% Sodium hydroxide (w/w)
Sodium Hydroxide solution - (1.0 N)- weigh 160g of the 50% sodium hydroxide in a 2000 mL volumetric flask and dilute to volume with deionized water.

PROCEDURE

1. Prepare a bench sheet.
2. Add 25 mL of the 1.0 N Sodium Hydroxide solution to each labeled scrubber and dilute to 100 mL with deionized water.
3. Weigh 10g or measure 10 mL of the waste into a labeled flask. Also set up a flask for a blank (deionized water), Reactive Cyanide LCS (add 2.5 mL of the 1000 mg/L cyanide standard and 7.5 mL DI water) and Reactive Sulfide LCS (add 10 mL of the sulfide standard).
4. Fill the addition funnel with 0.01N sulfuric acid (250 mL). Close the system and adjust the nitrogen flow to 60 mL/min. The flow may need to be adjusted to greater than 60 mL/min if a back pressure is present. Verify no fluid is rising in the dispersion tube.
5. With the nitrogen flowing, begin adding the sulfuric acid in the funnel to the flask. Start the 30-minute test period. Begin stirring while the acid is entering the flask. The stirring speed must remain constant. The stirring should not be fast enough to create a vortex.
6. After 30 minutes, close off the nitrogen and disconnect the scrubber.
7. Split the sample into aliquots, (~30 mL for cyanide and ~70 mL for sulfide). Deliver to the wet chemistry

department with a copy of the bench sheet.

- Analyze the cyanide by Method 9012A (LCHAT) and the Sulfide by Method 9034 (Titration). Substitute the following for Procedure Step 3 in SOP WL-051 (Sulfide Titration). The scrubber solution must be brought to a pH of 2 before proceeding. Test with pH paper. Stir or mix with minimal aeration when adjusting the pH.

CALCULATIONS

Total releasable HCN (mg/L or mg/kg) =
(instrument reading X 5 X DF) x 10

5 is a factor to correct for the water concentration factor included in the calibration.

Total releasable H₂S (mg/L or mg/kg) =
Concentration of H₂S in scrubber x 10

Concentration of H₂S in scrubber is calculated using the total sulfide formula in SOP-051. The mL of sample is equal to the mL of scrubber solution titrated.

The 10 factor for both cyanide and sulfide is =

$$\frac{(\text{volume of solution in scrubber (0.1 L)})}{(\text{weight of sample (0.01 kg)} \times 1800 \text{ seconds})} \times 1800 \text{ seconds}$$

REPORTING LIMITS

Reactive Cyanide is 250 mg/kg or mg/L
Reactive Sulfide is 80 mg/kg or mg/L.

QUALITY CONTROL

- Prep and analyze a blank with each batch of 20 or fewer samples. The concentration must be below the reporting limits or less than 5% of the concentration of any associated samples. If the blank does not meet this criterion, all samples in the batch must be reprepared.
- Prep and analyze an LCS with each batch of 20 or fewer samples. Perform an LCSD if insufficient sample is available to perform a batch duplicate. The LCS recovery limits are:

Reactive Sulfide: 47-106%
Reactive Cyanide: 1-20%

Sulfide - Concentration = True value of standard used for spiking the LCS (determined by titration of the standard)

Cyanide - Concentration = 250 mg/kg

If the LCS fails low for the above criteria, the entire batch of samples must be reprepared and reanalyzed. If the LCS fails high for the above criteria, samples may be reported if no results are above the regulatory limits

of 250mg/kg. In this case, the data should be flagged as estimated and a narrative should be included in the final report.

3. Duplicate one sample in each batch of twenty or fewer samples. The duplicate RPD must be ≤ 25 . If the duplicate does not meet this criterion, all samples in the batch must be reprepmed and reanalyzed unless it can be determined that the duplicate failure is the result of a sample that cannot be homogenized.

Sulfide - Concentration = True value of standard used for spiking the LCS (determined by titration of the standard)

Cyanide - Concentration = 250 mg/kg

METHOD PERFORMANCE

The operation of the system can be checked and verified by using the LCS. The LCS is processed as a sample and the % recovery is calculated. In this laboratory, 30 standards were analyzed for reactive cyanide with an average recovery of 7.58% and 30 standards were analyzed for reactive sulfide with an average recovery of 76.3%.

POLLUTION PREVENTION

See QAPP Section 13.2

WASTE MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
GC VOLATILES
STANDARD OPERATING PROCEDUREPROCEDURE GC-024
PAGE 1 of 9
EFFECTIVE DATE: 10/24/08
APPROVED BY: *MAP*
QA/QC APPROVED: *JDT*

SUBJECT SCOPE AND APPLICATION

RSK-175 is a method developed by the RS Kerr Research Laboratory, a division of the Environmental Protection Agency, to allow for the analysis of dissolved gases in groundwater. This analysis provides information in support of intrinsic bioremediation projects. This method is applicable to most dissolved gasses. Prior method validation shall be performed before the addition of other target compounds. This shall include an MDL study and demonstration of capability.

THEORY AND PRINCIPLE

A water sample is collected using a 44mL VOA vial. The vial is allowed to react at room temperature. Headspace is generated in the vial by injecting Helium and withdrawing an aliquot of the sample. The bottle is shaken vigorously for one minute and a sample of the headspace is analyzed using a GC equipped with a Flame Ionization Detector (FID) and a Thermal Conductivity Detector (TCD). Once the concentration in the headspace is determined, the concentration in the original water sample can be calculated using Henry's Law of Partial Pressures.

MATRIX Water

REFERENCES RSK SOP-175 Revision 0, 8/11/94, developed by the RS Kerr Research Laboratory, a division of the Environmental Protection Agency.

PRESERVATIVES Cool to 4°C ± 2°C
Samples should be collected in Level 3 44mL VOA vials. Every effort shall be made to minimize headspace in the vial prior to analysis. Samples should contain minimum of sediment.

HOLDING TIME 14 days from collection

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and the supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular,

approved gloves, safety glasses, and lab coats must be worn. In addition to other measures, solvents and chemicals must be handled in ventilated hoods.

The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed, solvents must be handled in ventilated hoods.

APPARATUS

HP 5890 Series II GC equipped with an FID and TCD

Target data acquisition system with sequence software connected to the GC using an A/D Interface

Gas-tight syringes - 50uL, 100uL, 500uL, and 10mL

SKC 1 liter Tedlar bags

REAGENTS

Label all containers and squeeze bottles with reagent ID, lot# and expiration date. All standards used must be pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. Label all working standards using completed standard labels.

1. Deionized water
2. HPLC water - bought commercially
3. Custom gas mixture - primary calibration
4. Custom gas mixture - second source
5. Helium, Grade 5
6. Hydrogen, Zero grade

INSTRUMENT
CONDITIONS

The following are the conditions that may be used during analysis. The analyst is allowed to optimize conditions as long as instrument conditions are not changed after initial calibration of the instrument is re-calibrated.

Starting temperature: 40°C
Hold for 1 minute
Ramp 30°C/minute to 120°C
Hold for 0.34 minutes
Run Time: 4.0 minutes
Injector temperature: 150°C
FID temperature: 300°C
TCD temperature: 140°C

NOTE: Because this analysis involves injection with a large bore needle, the septa shall be changed daily before use. If the septum needs to be changed more often because of bleed, document in equipment maintenance log. In addition, column liners should be changed often (generally every third use).

STANDARD

PREPARATION

Standards for this method can be prepared in two ways, by transfer to a Tedlar bag at the desired concentration (Tedlar bags should not be filled beyond approximately half-full), or by direct injection. If a Tedlar bag is used perform by placing a known volume of Helium in a Tedlar bag, and adding a measure volume of the pure gas as described in Table 2. The following chart contains a list of gases analyzed in this method, and concentration of the stock standard. Generally a direct injection method is used. Use Table 3 for spike amount.

Table 1 Stock Standards

Gas	Level (ppm V)	How Obtained
Methane	1000	Commercially prepared
Ethane	100	Commercially prepared
Ethene	100	Commercially prepared
Acetylene	1000	Commercially prepared
Propene (surrogate)	1000	Commercially prepared
Propane	1000	Dilute 0.5mL pure Propane in 500mL Helium
Carbon dioxide	20000	Dilute 10mL pure CO ₂ in 450mL Helium
Butane	1000	Dilute 0.5mL pure Butane in 500mL Helium
Iso-butane	100	Commercially prepared

Working standards are made depending on the analytes of interest. Generally Methane/Ethane/Ethene (MEE) are calibrated with Propene as a surrogate. Determine the calibration levels needed and dilute the standards with Helium using the following equation:

$$C_1V_1 = C_2V_2$$

Where C₂ is the concentration of the calibration standard in ppmV, V₂ is the total volume, C₁ is the concentration of the stock standard, and V₁ is the volume of the stock standard needed. See the Calculation section for determining the concentration in ug/L. An example is given in Table 2 showing the volume of each gas for the calibration standards analyzed.

Table 2 Working Standards

Analyte	Level 1 mL	Level 2 mL	Level 3 mL	Level 4 mL	Level 5 mL
Methane/Ethane/Ethene	5	25	50	100	150
Propene (surrogate)	5	25	50	100	150
Helium	490	450	400	300	0

Table 3 Direct Injection Spike Amount

Analyte	Level 1 uL	Level 2 uL	Level 3 uL	Level 4 uL	Level 5 uL
Methane/Ethane/Ethene	3	15	30	60	150
Propene (surrogate)	3	15	30	60	150

Table 4 Concentrations of Initial Calibration

Analyte	Conc Level 1		Conc Level 2		Conc Level 3		Conc Level 4		Conc Level 5	
	ppm V	ug/L								
Methane	10	1.09	50	5.45	100	10.9	200	21.8	500	54.4
Ethane	1	0.22	5	1.10	10	2.19	2	4.38	50	10.9
Ethene	1	0.29	5	1.44	10	2.89	20	5.78	50	14.5
Propene	10	6.39	50	32.0	100	63.9	200	128	500	319

RETENTION TIME
WINDOW

At set-up and after a column change, perform a retention time study by analyzing a standard (generally a the mid-point of the curve) over a minimum of 72 hours. Calculate 3 X the standard deviation (SD). Set the retention time of each analyte as the retention time in the initial calibration (last standard run) or the first CCV performed each day. The retention time window is set as the retention time $\pm 3SD$. Alternatively, if 3X SD is less than 0.03 minutes, a default retention time window of the retention time ± 0.03 minutes is used.

CALIBRATION

- Each analyte of interest for this method must be calibrated with a minimum of 3 points. Some projects may require more points. Inject 300uL of each calibration level prepared into the GC or the spike amount listed in Table 3 if direct injection is used. Calculate the RF and determine the RSD of the RF of all calibration points. The RSD must be <25% for the calibration to be valid. The analyst may drop points from either end to improve linearity, but must be aware that dropping points off the low end will affect the reporting limit and the minimum number of points for the project must remain. The acceptance criteria for initial calibration must be satisfied before analysis of samples can begin. Select projects may have additional or more stringent criteria that must be achieved for the applicable samples. See SOP GEN-019.
- After the initial calibration is evaluated, it must be verified by the analysis of an independent standard (initial calibration verification (ICV)). The ICV is prepared at a mid-level. Follow the procedure used for LCS. The ICV must be recovered at 75-125% to proceed with analysis. The retention time must be within the retention time window for each

analyte. Some projects have more stringent requirements for ICV criteria. See SOP GEN-019.

3. A Continuing Calibration verification (CCV) must be analyzed before any other sample every day an initial calibration is not performed, and at least every 12-hours thereafter to bracket samples. Determine the % Difference of the determined RF in the CCV and the average RF. The % Difference must be less than 20%. If the CCV at the end of a batch fails criteria, it may be re-analyzed once. If the analysis continues to fail, all samples analyzed since the previous passing CCV must be re-analyzed. Use the same standard used for the ICAL. The retention time must be within the retention time window for each analyte. Some projects have more stringent requirements for CCV criteria and/or frequency. See SOP-GEN-019.

PROCEDURE

1. Remove samples from the refrigerator and allow the samples to come to room temperature before analysis. Fill one 5mL gas-tight syringe with 4.5mL of Helium. Remove the syringe from the helium bag and insert into the Propene (surrogate) bag and continue to fill the syringe to the 5mL mark.
2. Insert this 5.0mL syringe and a second, empty 10mL gas-tight syringe through the septa of the sample VOA vial. As the 5.0mL syringe is injected, carefully remove 5.0mL of liquid with the 10mL syringe. Remove both syringes from the vial.
3. Shake the sample vigorously for 1 minute and allow the vial to sit for a minimum of three minutes before injection on the GC. This allows equilibrium to establish between the liquid and gas phase.
4. Using a 500uL syringe, remove 300uL of headspace and inject into GC injection port. Immediately press start in the GC and start on the A/D box.
5. Identify analytes based on the retention time of the peak. Concentrations are calculated using the equations in the Calculation section of this SOP.
6. If a sample requires dilution to bring the detected amount into calibration range, inject a smaller amount of sample using Table 5 as a guide. For dilutions $\geq 10X$, the surrogate is considered to be diluted out.

Table 5 Dilutions

Dilution	Injection Volume (uL)
1	300
2	150

5	100
10	60
20	30
300	20
50	12
100	6
200	3
3.00	2
600	1
900	0.5

QUALITY
CONTROL

1. Surrogate

- A. The surrogate for this method is Propene. Recovery criterion is 40-160%. Project specific recovery may be more stringent and will be listed in the LIMS and must be used for applicable samples. See SOP GEN-019. In some cases, Propene is requested as a target analyte. When this occurs no surrogate is used. If the surrogate is not recovered within criterion, check for matrix interferences. Reanalyze samples with failing surrogate recovery. If the surrogate fails high and there are no hits, report the sample result with a narrative. If the surrogate continues to fail in a sample, report the narrative. If the surrogate is biased high or low in the batch, perform corrective action and recalibrate the instrument.
- B. Spike all samples, calibration, and QC with 0.5mL/5mL Propene stock standard/total gas. Follow the steps in the procedure section.

2. Method Blank

- A. The method blank is prepped by filling a 44mL VOA vial with HPLC water and spiking with surrogate. Follow the procedure section for preparation and analysis.
- B. A method blank is run once per batch. For analysis to proceed, no target analyte may be found at a concentration greater than the reporting limit. Project specific criteria may be more stringent and will be listed in the LIMS. See SOP GEN-019.

3. Laboratory Control Standard

- A. The LCS is prepped in the same manner as a method blank with the addition of stock standards of the target analytes. It is spiked at a mid-level.
- B. The recovery must be 30-170%. Project specific recovery may be more stringent and will be listed in the LIMS. See SOP GEN-019.
- C. An LCS must be analyzed with every batch. Some projects may require the analysis of an LCSD.
- D. The LCS must pass to continue analysis of samples. If the LCS is above control limits and there are no target analytes present in the sample, the results may be reported with a narrative.

NOTE: For the LCS and the Method blank, if CO₂ is a target use deionized water instead of HPLC water.

4. Matrix Spike/Matrix Spike Duplicate

- A. Prep an MS/MSD in the same manner and at the same concentration as the LCS, except spike an aliquot of a sample, not reagent water.
- B. An MS/MSD are prepped with each batch. If there is insufficient sample to analyze an MSD, perform an LCSD.
- C. Use the same recovery criterion as the LCS.
- D. If the recovery fails, and the LCS recovery is within limits, check for co-elution and include a narrative.
- E. The precision must be calculated as RPD and must be <20%.

CALCULATIONS

The standards are prepped in ppmV. The results must be converted to ug/L to report. Target performs all calculations. An example is included on page 10.

$$P_{\text{gas}} = \frac{R_c}{1,000,000}$$

$$X_{\text{gas}} = \frac{P_{\text{gas}}}{H}$$

Where:

R_c = concentration in ppmV

X_{gas} = mole fraction of gas

H = Henry's constant for gas at 25°C

Table 6 Henry's Gas Constant (H)

Analyte	H at 25°C
Methane	41300
Ethane	30200
Ethene	11400
Acetylene	1330
Propene	5690
Propane	32700
Carbon Dioxide	2603
Butane	17150
Isobutane	14.66

Concentration of Gas (C_g):

$$C_g = (55.5 \text{ mole}) (X_g) (MW_{\text{g/mole}}) 1000 \text{ mg/g}$$

Where:

C_g = mg of gas in headspace

MW = molecular weight of gas (g/mole)

55.5 G mole = number of moles of water

Determine the concentration in the liquid phase by first calculating the density of gas in g/L:

$$D_g = \frac{MW \text{ (g/mole)}}{(22.4 \text{ L/mole}) (T/273)} \quad \text{Equation 3}$$

where:

22.4 L/mole = volume of 1 mole of gas at STP

T = temperature of headspace in °C/273

ML of analyte in headspace (A_H):

$$A_H = V_H P_G$$

Where:

V_H = headspace volume (mL)

Concentration of analyte in liquid phase (A_L):

$$A_L = (A_H/V_H)(D_G) * 100 \text{ mg/g} (1 \text{ L}/1000 \text{ mL}) \quad \text{Equation 4}$$

Where:

V_H = volume of water in L

Total concentration (T_C) of dissolved gas in water (mg/L):

$$T_C = (C_G/C_L) DF \quad \text{Equation 5}$$

$T_C (1000)$ = concentration in ug/L

The following equations are also used:

$$\text{MS \%REC} = \frac{\text{MS concentration} - \text{Sample concentration}}{\text{Spike added}} \times 100$$

$$\text{MSD \%REC} = \frac{\text{MSD concentration} - \text{Sample concentration}}{\text{Spike added}} \times 100$$

$$\% \text{ RPD} = \frac{\text{MS} - \text{MSD}}{\frac{\text{MS} + \text{MSD}}{2}} \times 100$$

$$\text{Surrogate Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

$$\text{Response Factor} = \frac{\text{Total Peak Area (counts)}}{\text{Amount of Analyte (ng)}}$$

$$\%RSD = (SD/X)100$$

where:

RSD = relative standard deviation

X = mean of the initial RF's for a compound

SD = standard deviation of the average RF's for a compound

REPORTING LIMITS

Reporting limits are defined by the low-level calibration standard. The low-level standard cannot be lower than the MDL and project specific requirements may define the relationship between the reporting limit and the MDL that must be achieved for the associated samples. See SOP GEN-019.

Methane	2.0ug/L
Ethane	1.0ug/L
Ethene	1.0ug/L
Isobutane	0.5ug/L
Acetylene	2.0ug/L
Propane	5.0ug/L
CO2	600ug/L
Butane	5.0ug/L

MANUAL INTEGRATION

Manual integrations are performed extensively for this method. Perform and document manual integrations as required by SOP QA-010

METHOD PERFORMANCE

An MDL study is performed as required by SOP QA-009. Each analyst using this method must demonstrate capability before reporting results and yearly thereafter, either by having acceptable recovery and precision for 4 LCS, or achieving acceptable results on a PE or other blind study.

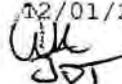
POLLUTION PREVENTION

See QAPP Section 10.2

WASTE MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC
WET LAB
STANDARD OPERATING PROCEDURE

PROCEDURES: WL-033
PAGE: 1 OF 4
EFFECTIVE DATE: 12/01/2008
APPROVED BY: 
QA/QC APPROVED: 

SUBJECT SCOPE AND APPLICATION

This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes. Sulfide reacts with N,N-dimethyl-p-phenylenediamine sulfate to form Methylene blue, a dye that is measured at a wavelength of 665 nm. The intensity of the blue color is proportional to the sulfide concentration. Acid insoluble Sulfides are not measured by this method. Copper sulfide is the only common sulfide in this class.

REFERENCE SM 4500 S D/HACH 8131

MATRIX Water

PRESERVATIVE Add Zinc Acetate and Sodium Hydroxide to a pH >9, Cool 4°C
Avoid excess agitation or prolonged exposure to air.

HOLDING TIME 7 days

DEFINITIONS See SOP GEN-016

SAFETY Use safety glasses and gloves. All other safety precautions should be considered.

APPARATUS HACH 2800 Spectrophotometer set at 665 nm
Timer
10 mL cells
Plastic cups
Pipette
Graduated cylinders
Mechanical pipet and tips

REAGENTS HACH Sulfide Reagent #1 - (N,N-dimethyl-p-phenylenediamine solution)
HACH Sulfide Reagent #2 - (potassium dichromate)
Sodium Sulfide crystals - Two lots - prepare a stock standard at ~ 1000 mg/L by dissolving 7.6 g of the crystals in deionized water and diluting to a final volume of 1000 mL. The pH should be >9 to <11. Adjust with sodium hydroxide. The true value of the stock standard is determined by the titration method - See SOP WL-051. The true value is determined each day of use. The standard can be used for one month.

INTERFERENCES Color and turbidity may interfere with observations of color or with photometric readings. Do not shake sample due to the volatility of Sulfide. Strong reducing substances (Sulfite, Thiosulfate, and Hydrosulfite) interfere by producing the blue color or preventing the development. Sulfide in high concentrations may inhibit the full color development and require dilution. Some Sulfide loss may occur when the sample is diluted.

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PROCEDURE

CALIBRATION

1. Recall user program 952.
2. Set wavelength to 665 nm.
3. Prepare a calibration curve or verify an existing curve.
4. Prepare a calibration curve and prepare calibration standards. The standards are prepared from the working standard or a dilution of the working standard. The standards may be serially diluted from higher concentration standards. Prepare 100 mL of each standard: 0.02, 0.1, 0.5, 1.0, 1.5, and 2.0 mg/L.

The blank for the curve is deionized water with Zinc acetate and Sodium hydroxide to a pH of >9.

5. Measure 25 mL of deionized water in a sample cell and place in the sample slot. Zero the instrument.
6. Measure 25 mL of each calibration standard and 25 mL of deionized water (calibration blank) and pour into plastic cups.
7. Add 1 mL of Reagent #1 to the calibration blank. Swirl to mix. Add 1 mL of Reagent #2 to the calibration blank. Swirl to mix. A pink color will develop, then the solution will turn blue if Sulfide is present. Allow a five-minute reaction time. Set the timer.
8. Transfer the contents of the blank into a sample cell. Wipe the cell and place in the cell holder. Read on the instrument.
9. Repeat steps 7 and 8 for all standards.
10. The correlation coefficient must be 0.995 for a valid curve.
11. An existing curve can be verified by preparing a 1.0 mg/L standard from the working standard then following steps 6-8 above. The recovery must be 90-110% to continue the analytical run.
12. A new curve must be performed when the acceptance criteria is not met or at least annually.

SAMPLE ANALYSIS

1. Build the sample batch in the LIMS and print the batch worksheet.
2. Mark the sample level on the container. Decant the supernatant (to level of precipitate or flock). Refill to the line with deionized water. The flock is Zinc sulfide. This is performed to eliminate interference from Sulfite, Thiosulfate, Iodide and many other soluble substances.
3. Transfer 25 mL of the first sample to a cup. Minimize shaking of the sample.

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5. Repeat for all samples.
6. Dilute any sample with a concentration greater than the high concentration standard.
7. Print the raw data. Complete all information on the batch cover sheet. Download data to the LIMS.

SAMPLE ANALYSIS FOR INSOLUBLE SULFIDE

1. Build the sample batch in the LIMS and print the batch worksheet.
2. In a 200mL separatory funnel, measure 200mL of sample.
3. To each funnel add 0.4 mL of AlCl and 0.4 mL of NaOH.
4. Mix vigorously for 1 minute.
5. Decant off approximately 100mL of the clean layer.
6. Add 0.15mL of Zinc acetate solution and 0.1 mL of NaOH.
7. Mix vigorously for 1 minute.

CALCULATIONS

Sulfide mg/L = Instrument Reading x Dilution Factor

QUALITY CONTROL

1. Prepare an independent standard (prepare from stock not used for calibration) at a concentration of 1 mg/L. Analyze as a sample immediately after the calibration. The recovery must be 90-110%
2. Analyze one method blank with each batch of 20 or fewer samples. The concentration should be less than $\frac{1}{2}$ the reporting limit.
3. Analyze one LCS with each batch of 20 or fewer samples. Prepare at a concentration of 1.0 mg/L. The recovery must be 90-110%. A LCSD may be performed if required for the project or if there is insufficient sample for a duplicate or MSD. If the LCS recovery is high and there is no sulfide detected in the samples, the data may be reported with a narratives
4. Duplicate one sample in each batch of 20 or fewer samples. The RPD should be ≤ 25 . The RPD limit for a MS/MSD should be ≤ 25 . Document failures in the case narrative.
5. Perform a matrix spike (MS) on one sample in each batch of 20 or fewer samples. The recovery should be 75-125%. A matrix spike duplicate (MSD) may be performed if required for the project. The control limits are the same as for the MS. Document failures in the narrative.
6. Analyze a CCV (1 mg/L) at the beginning of the run, after every 10 samples and at the end of the run. The recovery must be 90-110%. If a recovery is high and there is no sulfide in samples bracketed by the CCV, the data may be reported with a narratives

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concentration must be less than $\frac{1}{2}$ the reporting limit.

NOTE: The concentration of stored stock standards is labeled as TBD (to be determined) due to the concentration being determined on a daily basis.

REPORTING LIMITS 0.02 mg/L

METHOD

PERFORMANCE The precision has not been determined. The accuracy is about $\pm 10\%$.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

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GULF COAST ANALYTICAL LABORATORIES, INC.
WET LAB
STANDARD OPERATING PROCEDURE

PROCEDURE: WL-043
PAGE: 1 OF 9
EFFECTIVE DATE: 10/08/2008
APPROVED BY: *MAP*
QA/QC APPROVED: *JDT*

SUBJECT SCOPE AND APPLICATION

To identify quantities of organic carbon compounds in drinking water, surface and saline waters, domestic and industrial waste. Used to assess the potential oxygen-demanding load of organic material on a receiving stream. This procedure measures total and inorganic carbon. Organic carbon is determined by calculating the difference between total carbon and inorganic carbon.

MATRIX Water

REFERENCES Standard methods, 18th ed. method 5310B
SW-846, 9060A
Instrument manual

SAMPLE COLLECTION
& HANDLING

Samples may be collected in glass bottles. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis and laboratory fortified matrix analysis, and minimize waste disposal. The recommended quantity for water is 50 mL.

CHAIN OF CUSTODY See QAPP 11.2-11.3

PRESERVATION Cool 4°C, H₂SO₄ or HCL to pH <2.

HOLDING TIME 28 days

DEFINITIONS See SOP GEN-016

SAFETY

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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents and chemicals must be handled in ventilated hoods.

APPARATUS

Shimadzu TOC-5050
ASI-5000 TOC Autosampler
Shimadzu TOC-V_{CSH}

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ASI-V Autosampler
40 mL VOA vials
UHP air
Dixie cups
10 mL pipettes class A
10 mL pipettes plastic
100 and 200 mL volumetric flask
Shimadzu autosampler vials
0.50 mL autopipettor (MLA) and tips

REAGENTS &
STANDARDS

Deionized water (DI)

Hydrochloric acid - preservative

Stock standard - organic carbon (TC) (1000 mg/L),
commercially prepared. Used to prepare the calibration curve.

Stock standard - organic carbon (TC) (1000 mg/L), commercially
prepared. Independent from the source used for calibration.

Stock standard - inorganic carbon (IC) (1000 mg/L),
commercially prepared. Used to prepare the calibration curve.

Stock standard - inorganic carbon (IC) (1000 mg/L),
commercially prepared. Independent from the source used for
calibration

High Purity Compressed Air cylinder - carrier gas

25% Phosphoric Acid - 25mL Phosphoric acid brought up to 100
mL with DI water

0.3N NaOH - 2.4g NaOH brought up to 200 mL with DI water.

INTERFERENCES

In determining DOC avoid any contact with organic material
that may contaminate a sample. Avoid contaminated glassware,
plastic containers and rubber tubing.

STANDARD PREPARATION

Record preparation of all standards in the standards log.

Calibration Standards: Prepare standards from the calibration
stock (TC & IC) at concentrations of 1, 15, 30, 60, 100 mg/L
in 100 mL volumetric flasks. Dilute to volume with deionized
water. Lower concentration standards may be prepared from the
higher concentration standards.

ICV Standard: Prepare a 50 mg/L (TC & IC) from the independent stock standard. Prepare in a 200 mL volumetric flask and dilute to volume with deionized water.

Continuing Calibration Verification Standard (CCV): Prepare a 50mg/L standard from the calibration stock (TC & IC). Prepare in 200 mL volumetric flask and dilute to volume with deionized water.

Laboratory Control Standard: Prepare a 50mg/L standard from the calibration stock (TC & IC). Prepare in a 200 mL volumetric flask and dilute to volume with deionized water.

Matrix Spike/Matrix Spike Duplicate: For TOC-5050, spike 9.5 mL of the sample selected for spiking by adding 0.5 mL of the 1000 mg/L independent TC standard. For TOC- V_{CSH} , add 5.0 mL of the 1000 mg/L independent TC standard and dilute to a final volume of 100 mL with the sample selected for spiking in a 100 mL volumetric flask. Both techniques yield a spike concentration of 50 mg/L.

PROCEDURE
FOR TOC-5050

INSTRUMENT START-UP

1. Turn power on with white switch on right side.
2. Check compressed air supply. If quantity of regulator is below 250 psi then replace bottle, as 200 psi minimum is required for one day's operation.
3. Set carrier gas pressure at 150. The sparge gas should be off.
4. Check reagent bottles inside the instrument.
 - The bottle on the left (IC reagent reservoir) contains 25% Phosphoric acid. Make sure volume is between 75 - 125 mL.
 - The bottle on the right (Humidifier) contains 0.3N NaOH. The liquid level should never fall below the lower line, nor should it be filled above the upper line.
5. Make sure the reservoir on the autosampler is filled with DI water for rinsing.

CALIBRATION

Pour standards in the autosampler vials and load in the

selected positions on the autosampler.

1. Select Edit: Insert standard/calibration curve list/new.
2. Title: enter year/month/date (TC or IC).
3. File name: year/month/date (TC or IC) (ex: 030918TC).
4. Enter the required information: calculation method: linear regression without zero shift; range: 5; select analysis type: TC or IC; Max SD: 400. TC injection volume is 27 uL.
5. Go to the data tab: enter the required information: Sample ID: Level 1-Level 5; Sample ID: enter concentration (ex: 1.0); Vial: enter autosampler position; Concentration: enter concentration; No of injections: enter 4; Max number of injections: (5)* autoset.
6. Select OK; File name appears: Select OK
7. Repeat for IC. IC injection volume is 33 uL.
8. Select measurement/start. Select continue to run. OK.
After completion of the calibration procedure, review the calibration summary. The correlation coefficient must be ≥ 0.995 . If this criterion is not met, review each standard to determine if the problem appears to be associated with a single standard. If a single standard appears to be the problem, prepare a new standard and re-analyze the calibration curve. If the problem is not associated with a single standard, prepare all new standards or perform instrument maintenance and re-analyze the calibration curve.
9. A new curve must be prepared when the calibration stock standard expires, the CCV fails, or at least annually.
10. After each initial calibration, analyze the ICV standards to verify the calibration with an independent source standard. The recoveries must be 90-110% to proceed with sample analysis.
11. Each analytical sequence is started with the analysis of a CCV (TC & IC) at concentrations 50 mg/L. CCV's (50mg/L) for both TC and TIC are analyzed after every 10 samples positions. The recovery for all TC CCV's should be 90-110%. The recovery for the initial TIC CCV should be 90-110% with all subsequent TIC CCV's falling within the

range of 88-126%.

SAMPLE PREPARATION AND ANALYSIS

1. Go to Edit: Insert Sample; Select method and open. Click on the TC tab; Select the most recent TC calibration and click OK. Click on the IC tab; select the most recent IC calibration and click OK.
2. Create the sample batch in the LIMS. Print a Batch Sample Worklist (BSW).
3. Go to Edit: Auto Generate; click edit method and choose method; OK. Enter the start vial (1) and the ending vial (based on the number of samples on the BSW). Click OK.
4. Highlight the sample name and sample ID cells, select cut (scissors) to allow entry of the sample IDs. Place the cursor on Vial 1. Build the batch by scanning the barcode for each sample on the BSW and entering the information on the spreadsheet. Scan each sample twice (TC & IC). Insert the CCV and CCBs in the correct locations. Save as year/month/day/run# (03092301). If analyzing samples for method 9060, revise the number of injections to 4 for the samples and associated QC.
5. Homogenize samples and pour into autosampler vials. Exclude particles that can clog the syringe. Place in the correct position on the autosampler. If samples are analyzed for dissolved organic carbon and have not been field filtered, filter an unpreserved aliquot of sample through a 0.45 μ m filter and then add the appropriate preservative. Deionized water must be filtered as the method blank.
6. Pour deionized water in autosampler vials and place in the positions for the method blank and CCBs. Pour the LCS and LCSD (if required) in autosampler vials and place in the assigned positions on the autosampler. Pour the appropriate CCVs into autosampler vials and place in the assigned positions on the autosampler. Prepare the MS and MSD (if required), pour into vials and place in the correct positions on the autosampler. Pour a second aliquot of the batch duplicate into a vial and place in the correct position on the autosampler.
7. Go to start; select continue to run.

8. At the completion of the run, review all QC against the acceptance criteria. Rerun any samples that exceed the calibration range of the instrument (after instrument dilution). The instrument will automatically rerun with a reduced volume if a concentration above the high level standard is detected. Verify that carryover has not occurred after analysis of a high concentration sample. Rerun samples if suspected carryover has occurred.
9. If batch is acceptable, download data to the LIMS.

PROCEDURE
FOR TOC- V_{CSH}

INSTRUMENT START-UP

1. Turn power on with white switch on the front.
2. Check compressed air supply. If quantity of regulator is below 250 psi minimum is required for one day's operation.
3. Set carrier gas pressure at 150. The sparge gas should be off.
4. Check reagent bottles on the left side of the instrument. The IC reagent reservoir contains 25% Phosphoric acid and the dilution reservoir contains DI water.
5. Make sure the reservoir on the autosampler is filled with DI water for rinsing.

CALIBRATION

Four standards into 40 mL VOA vials and load in the selected positions on the ASI-V autosampler.

1. Select "Open", then double click "calibration template"
2. Select "Edit", then "Clear measured data", then "all".
3. Select "Connect" and wait for instrument.
4. Select "Start".
5. After completion of the calibration procedure, review the calibration summary. The correlation coefficient must be ≥ 0.995 . If this criterion is not met, review each standard to determine if the problem appears to be associated with a single standard. If a single standard appears to be the problem, prepare a new standard and re-analyze the calibration curve. If the problem is not associated with a single standard, prepare all new standards or perform instrument maintenance and re-analyze the calibration curve.
6. A new curve must be prepared when the calibration stock standard expires, the CCV fails, or at least annually.

7. After each initial calibration, analyze the ICV standards to verify the calibration with an independent source standard. The recoveries must be 90-110% to proceed with sample analysis.
8. Each analytical sequence is started with the analysis of a CCV (TC & IC) at concentrations 50 mg/L. A CCV 50 mg/L (TC) is analyzed after every 10 samples (positions). The recovery for all CCVs must be 90-110%. All reportable data must be bracketed by acceptable CCVs.

SAMPLE PREPARATION AND ANALYSIS

1. Create sample batch in LIMS. Print a batch sample worklist (BSW).
2. Select "New", then "OK".
3. Select "Connect" and wait for the instrument.
4. Highlight the "1" in the top left corner of the spreadsheet. Right click and select "Insert Multiple Samples".
5. Click on the "." next to the method blank and select "TOC3.met", then "OPEN". Select "Next". If analyzing samples for method 9060, select "TOC4.met" instead.
6. Enter the number of samples desired for the batch. Choose the appropriate start vial position. Select "Finish". Select "OK".
7. Highlight "Sample name" and "Sample ID" columns on the spreadsheet and right click and select "Cut".
8. Place the cursor in the sample name column. Build the batch scanning the barcode for each sample on the BSW. Insert CCV's and CCB's in the correct locations.
9. Homogenize samples and pour into 40 mL VOA vials. Exclude particles that can clog the syringe. Place in the correct position on the autosampler. If samples are analyzed for dissolved organic carbon and have not been field filtered, filter an unpreserved aliquot of sample through a 0.45 um filter and then add the appropriate preservative. Deionized water must be filtered as the method blank.

NOTE: Many samples will have already been collected in the 40mL VOA vials.

10. Pour deionized water in 40 mL VOA vials and place in the positions for the method blank and CCBs. Pour the LCS and LCSD (if required) in autosampler vials and place in the assigned positions on the autosampler. Pour the appropriate CCVs into autosampler vials and place in the assigned positions on the autosampler. Prepare the MS and MSD (if required), pour into vials and place in the correct positions on the autosampler. Pour a second aliquot of the batch duplicate into a vial and place in

the correct position on the autosampler.

11. Select "Start", then "Save". The instrument automatically names the file.
12. Make sure "Keep running" is selected, and then select "Start".
13. At the completion of the run, review all QC against the acceptance criteria. Rerun any samples that exceed the calibration range of the instrument (after instrument dilution). The instrument will automatically rerun with a reduced volume if a concentration above the high level standard is detected. Verify that carryover has not occurred after analysis of a high concentration sample.
14. If the batch is acceptable, download the data to the LIMS.

MAINTENANCE

1. Daily - verify proper volume solutions in instrument
2. First injection - check for air bubbles in syringe.
3. First injection - verify syringe does not leak.
4. Check Halogen scrubber - if dark in color, replace.
5. If bubbling in drain pot is present, change membrane filter.

CALCULATION

TOC or DOC mg/L = Total Carbon - Inorganic Carbon

Total Carbon = instrument reading x dilution factor

Inorganic Carbon = instrument reading x dilution factor

QUALITY CONTROL

1. Analyze one method blank with each batch of 20 or fewer samples. The concentration must be $< \frac{1}{2}$ the reporting limit or less than 5% of the TOC concentration in the associated samples. If the method blank criteria are not met, all samples in the batch must be reanalyzed.
2. Analyze a LCS and LCSD (if required) with each batch of 20 or fewer samples. Recovery must be 80-120. If the LCS criteria are not met, all samples in the batch must be reanalyzed.
3. Perform a MS and MSD (if required) on one sample in each batch of 10 or fewer samples. The recovery must be 75-125%. If the recovery is outside the control limits and the LCS/LCSD recoveries are acceptable it may indicate matrix interference. Spiking a diluted aliquot of the sample may verify the presence of interference.
4. Duplicate one of every 20 samples. The RPD must be ≤ 25 for concentrations > 5 times the reporting limit. If the RPD

is exceeded include a narrative with the data.

5. ICV and CCV criteria are included in the calibration section. Analyze a CCB (deionized water) after each CCV. The concentration must be $< \frac{1}{2}$ the reporting limit or less than 5% of the TOC concentration in the associated samples. If the CCV or CCB criteria are not met, all samples that are not bracketed by passing CCV's and CCB's must be reanalyzed.
6. Verify 4 injections were used for method 9060.
7. Verify that any injections that exceed the maximum CV of 2.0% have been reanalyzed by the instrument and excluded from the final concentration. If not, the sample must be reanalyzed.

REPORTING LIMIT 1.0 mg/L

METHOD

PERFORMANCE

Twenty-eight analysts in twenty-one laboratories analyzed distilled water solutions containing exact increments of oxidizable organic compounds, with the following results:

Increment as TOC mg/liter	Precision as Standard Deviation TOC, mg/liter	Accuracy as Bias, %	Accuracy as Bias, mg/liter
4.9	3.93	+15.27	+0.75
107	8.32	+1.01	+1.08

Precision: The difficulty of sampling particulate matter on unfiltered samples limits the precision of the method to approximately 5-10%. On clear samples or on those that have been filtered before analysis, precision approaches 1 to 2% or ± 1 to 2 mg carbon/L, whichever is greater.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
GENERAL
STANDARD OPERATING PROCEDURES

PROCEDURE: GEN-009
PAGE: 1 OF 4
EFFECTIVE DATE: 05/19/09
APPROVED BY: *MAP*
QA/QC APPROVED: *JBT*

SUBJECT SCOPE AND APPLICATION

Gulf Coast Analytical Laboratories, Inc. - Baton Rouge uses this procedure to ensure the collection, storage, transportation and disposal of waste is in compliance with 40 CFR parts 260/261/262 (EPA), 29 CFR (OSHA), and 49 CFR (DOT). The types of waste generated include chemicals, reagents, solvents, standards, and samples.

SAFETY

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 personal protective equipment and spill clean-up procedures PRIOR TO the use of any chemical. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

PROCEDURE

1. Laboratory waste is segregated by laboratory personnel into waste streams, which have been established by the Regulatory Compliance Officer. The waste streams are determined by analysis of the waste and through process knowledge. All laboratory waste should be disposed of in the proper container. No laboratory waste shall be placed in regular trash containers for disposal. The waste is placed into 55-gallon drums, which are located in Satellite Waste Accumulation Areas. These Satellite Waste Accumulation Areas are labeled with the appropriate placards as to be easily identified. A single Satellite Waste Accumulation Area cannot contain more than 55 gallons of any one waste stream. When a 55-gallon drum in a Satellite Waste Accumulation Area is full, the Regulatory Compliance officer is contacted to move the drum to the Central Accumulation Facility where the start accumulation date begins. The laboratory has 90 days from the start accumulation date to dispose of the drum. The

laboratory will make every effort to minimize waste and reclaim materials whenever possible.

2. A waste is considered hazardous if:
 - A) The waste material is listed as hazardous in 40 CFR Part 261.30-261.33.
 - B) The material exhibits any of the characteristics of hazardous waste (ignitability, corrosivity, reactivity, or exceedance of a regulatory limit on a TCLP compound).
 - C) The waste listed in A or B above is not excluded by any provisions under the Resource Conservation and Recovery Act.

3. A waste is considered an acute hazardous waste if it is identified as such in 40 CFR Part 261.31, 261.32, or 261.33. The Regulatory Compliance Officer will be responsible for characterizing and disposing of waste. Samples submitted to the laboratory for analysis are excluded under CFR Part 261.4(d) provided that the samples are being transported to or from the laboratory, are being analyzed, are being held for analysis, or are being maintained in custody for legal reasons. RCRA regulations will apply 90 days after the login date of a sample (40 CFR Part 261.4). A sample may be held up to one year for future analysis under 40 CFR Part 261. General procedures followed by the laboratory will include transferring the samples to the Central Accumulation Area within 90 days.

4. Sample Disposal
Following refrigerated storage in the laboratory, the sample custodian will routinely stage completed samples for disposal. After removing samples, the sample custodian will notify the Regulatory Compliance Officer that the samples are ready to be taken to the Staging Area.

Following staging, sample disposal will begin. Samples are segregated based on sample matrix and characteristics. Water samples are collected in 55-gallon tight-head poly drums and labeled according to sample characteristics and waste stream. This disposal is performed below the canopy hood in the disposal trailer. Solid samples are collected in 55-gallon open top steel drums. The drums are labeled according to characteristics and waste stream. Personnel trained to perform this disposal wearing the proper PPE including but not limited to: Tyvek suits, full-face respirators, gloves, hearing protection. This activity

is performed under the supervision of the Regulatory Compliance Officer.

5. All drums are stored on the secondary containment pallets installed in the disposal trailer. Whenever personnel occupy the trailer, the ventilation system at the rear of the trailer must be activated. This will eliminate the build-up of possible dangerous vapors in the trailer.

6. Drum Labeling and Identification

All drums in the disposal trailer must be labeled with the following information:

Only one (1) date will be placed on drums.

Waste from satellite areas and solvent drums are dated when completed and moved to the Central Accumulation Area.

All drums that are used for sample disposal (water samples, solids, and glass jars) in the disposal trailer will be dated when the disposal process begins.

Proper descriptive labels will be placed on all drums.

7. Waste disposal service is provided by various approved vendors who come to the facility and label, placard, and manifest the drums according to 49 CFR before transporting to their facility. These hazardous waste drums will be incinerated, land filled, treated, or reclaimed depending on their character. The original completed manifest is mailed from the vendor to the laboratory to show the final disposal of the waste drums. The Regulatory Compliance Officer maintains these records. Copies of all manifests are mailed to LADEQ to report the disposal activities of the facility.
8. A waste inspection checklist will be performed on a weekly basis (See Attachment 1). These checklists will be maintained in a bound logbook.

STANDARD OPERATING PROCEDURE

Quantitative Polymerase Chain Reaction (qPCR)

SOP Number: DNA-qPCR

Revision Number: 1.0 FINAL

Revision Date: 01/10/06

Effective Date: 01/10/06

Reviewed By: _____ Date _____
Dora Ogles
DNA Lab Director

Reviewed By: _____ Date _____
Aaron Peacock
QA Coordinator

Reviewed By: _____ Date _____
Greg Davis
President/Lab Director

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Quantitative Polymerase Chain Reaction

Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

SOP No. DNA-qPCR

Revision No. 1.0 FINAL

Revision Date: 01/10/06

Effective Date: 01/10/06

Page: 3 of 7

PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the activities involved in the preparation, handling, documentation, and analysis of samples collected from a variety of matrices (water, soil, bio-traps, etc) to enumerate target populations using quantitative Real-Time polymerase chain reaction or qPCR.

1.0 SCOPE AND APPLICATION

Quantitative Real-Time polymerase chain reaction (qPCR) is used to enumerate target populations of microorganisms. Microbial Insights, Inc. (MI) uses qPCR to target the following organisms associated with the remediation of a priority pollutants such as Tetrachloroethene (PCE) and Methyl *tert*-butyl ether (MTBE).

Q-Target	MI Code or CAS Number	Real-time assay
<i>Dehalococcoides spp.</i>	qDHC	Taqman
<i>Dehalobacter spp.</i>	qDHB	SYBR green
<i>Desulfuromonas spp.</i>	qDSM	Taqman
<i>Desulfitobacterium spp.</i>	qDSB	SYBR green
Methanogen (mcrA gene)	qMGN	SYBR green
Iron and Sulfate Reducing Bacteria	qIRBSRB	Taqman
Dissimilatory Sulfite Reductase	qDSR	SYBR green
Methane Oxidizing Bacteria	qMOB	SYBR green
PM1	qPM1	Taqman
Universal Baceteria	qEBAC	Taqman
Soluble Methane Monooxygenase	qsMMO	SYBR green
Ammonia Oxidizing Bacteria	qAOB	Taqman
Anaerobic Toluene	qbssA	Taqman
<i>Geobacter spp.</i>	qGEO	Taqman
Denitrifying Bacteria (nirS and nirK)	qDEN	SYBR green
Catechol Dioxygenase	qCAT	SYBR green
Toluene Monooxygenase	qRDEG	SYBR green
Toluene Monooxygenase	qRMO	SYBR green
Xylene Monooxygenase	qTOL	Taqman
Naphthalene Dioxygenase	qNAH	Taqman
Phenol Monooxygenase	qPHE	SYBR green
Acetogens(FTHFS gene)	qACE	SYBR green
Alkane Monooxygenase	qALKb	Taqman
Butane Monooxygenase	qBMO	Taqman
Toluene Dioxygenase	qTOD	Taqman

2.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

Samples are collected following the standard operating procedure developed by Microbial Insights, Inc (MI). DNA extraction and RNA extraction are performed following the extraction standard operating procedure developed by Microbial Insights, Inc. DNA is stored in 1/10 Tris-EDTA(TE) at 4°C while RNA is stored in RNA Storage solution at -20°C. RNA samples are converted to cDNA following (SOP-RNA ext) prior to the quantitative PCR analysis.

3.0 EQUIPMENT AND SUPPLIES

- 96-well plate (Applied Biosystems).
- Micropipettes (Gilson P-10, P-100, P-1000) and 10 ml pipettes, and appropriate sterile tips (ART). [see calibration listed in calibration log]
- Applied Biosystems SDS 7000 or 7300 instrument. [see calibration listed in calibration log]
- Sterile 0.5ml thin wall strip tubes and optical clear caps (Applied Biosystems)

4.0 REAGENTS AND POSITIVE CONTROLS

- 4.1 TaqMan DNA polymerase and master mix for Taqman assays (Applied Biosystems)
- 4.2 Sterile filtered (0.2mM) nanopure, organic-free, deionized water.
- 4.3 Forward and Reverse primers/Probe: these are gene/assay specific (IDT Technologies primers, Applied Biosystems FAM/TAMRA probes). See notes section for primer handling. Final concentration of probe and primers: [REDACTED] or adjusted as needed for each primer set. [REDACTED]
- 4.4 Positive and Negative Control DNA: DNA is purchased from ATCC or extracted and purified in-house from pure isolates. The amount of DNA used in all assays is 3µl. For negative controls, water is added in place of template DNA.
- 4.5 [REDACTED] for SYBR green assays ([REDACTED])
- 4.6 [REDACTED] for SYBR green assay
- 4.7 [REDACTED]
[REDACTED]
[REDACTED]

5.0 PROCEDURE

5.1 Preparation of Stock Primer

5.1.1 It is necessary to resuspend and dilute our primers (IDT Technologies) before we can use them for our PCR reaction. This procedure is done when new primers are received and both working stocks and solution stocks should be prepared at this time. The label on the tube will provide the oligo length and concentration.

5.1.2 To the stock tube, aseptically add 1ml of sterile PCR dH₂O and vortex the tube for 1 minute to resuspend the primer. Centrifuge for 1 minute at 14,000 rpm to remove any solution from the lid then allow the tube to sit at room temperature for 5 minutes to ensure complete dissolution. This provides the solution stock.

5.1.3 To make the working stock to be used in each reaction, add the amount of nanomoles of the stock solution (listed on the stock tube) to a clean 1.5ml tube and bring this volume up to 1ml with sterile dH₂O.

5.2 Mixing Reagents-Taqman Assays

5.2.1 Prepare a master mix including the appropriate amounts of each of the following: PCR water, Taqman Universal PCR mix, Primers (forward and reverse) and Probe. The appropriate amounts are listed in the MI primer list (primer.xls) prepared for each primer/probe combination.

5.2.2 Add [REDACTED] to each well of the 96 well plate or strip tube. Add [REDACTED] template DNA/cDNA to the appropriate wells. Add [REDACTED] positive control DNA to the appropriate wells. Add [REDACTED] PCR water to the negative control wells.

5.2.1 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.2.2 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see table1).

5.2.3 Upon completion of the run, export the data to an Excel worksheet to determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules.

5.2.4 Standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the R² of each equation must be at least 0.95.

5.3 Mixing Reagents-SYBR green Assays

Quantitative Polymerase Chain Reaction

Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

SOP No. DNA-qPCR

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5.3.1 Prepare a master mix including the appropriate amounts of each of the following ingredients: PCR water, [REDACTED], PCR Primers (forward and reverse), [REDACTED] Reaction Buffer, [REDACTED].
[REDACTED] The appropriate amounts are listed in the excel sheets prepared for each primer combination.

5.3.2 Add [REDACTED] of master mix to each well of the 96 well plate or strip tube. Add [REDACTED] template DNA/cDNA to the appropriate wells. Add [REDACTED] positive control DNA to the appropriate wells. Add [REDACTED] PCR water to the negative control wells.

5.3.3 Spin the plate in the centrifuge at 3600 rpm for 2 minutes to be sure all mix is in the bottom of the tube and that no bubbles are present.

5.3.4 The plate of samples is then placed in either the ABI 7000 or 7300. The conditions of each assay will vary depending upon the primer/probe combination (see DNA primers xls data sheet)

5.3.5 Upon completion of the run, export the data to an Excel worksheet, then determine the number of target molecules, using an equation derived from standard curves of known numbers of target molecules. The standard curves are developed from a serial dilution of a known concentration of the positive control organism. At least 4 dilutions will be used for each curve and the R² of each equation must be at least 0.95.

6.0 QUALITY CONTROL

6.1 For quality control of the amplification process, positive and negative controls are always performed. The extraction blank from each set is also amplified to ensure no cross contamination of samples or solutions during the extraction process.

6.2 Ongoing calibration checks and maintenance of the instruments must be documented in the appropriate logbook.

6.3 The Laboratory Director has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience to process the samples.

6.4 All deviations from this SOP must be documented in a Nonconformance Report (NCR) or using an equivalent system (i.e., database).

7.0 ANALYTICAL REPORT

7.1 All data regarding quantities are entered into a data report using the laboratory information management systems (LIMS).

Quantitative Polymerase Chain Reaction
Matrix: Variety (Water, Soil/Solid, Bio-Trap, etc)

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7.2 All documentation pertinent to a project will be placed in the "project folder" identified with the unique LIMS Project number. Hardcopy records, data sheets, and soft copy reports will be stored for a period of three years.

8.0 DOCUMENT CORRECTIONS

Changes or corrections on any laboratory documentation will be made by crossing out the erroneous item with a single line and initialing (by the person performing the correction) and dating the correction. The original item, although erroneous, must remain legible beneath the cross-out line. The new information should be written clearly above the crossed-out erroneous item. All information will be recorded using black or blue indelible ink.

9.0 TABLES, DIAGRAMS, AND FLOWCHARTS

See DNA primer xls sheet for primer conditions.

Document Short Name**Document Number****Administrative:**

Chain of Custody	MI SOP COC
Sample Reception	MI SOP SampleReception
Glassware	MI SOP Glassware
Bottle Order	MI SOP BTLP01-06
	MI SOP Waste Disposal

Culture:

Anaerobic Chamber	MI SOP Anaerobic Chamber
Anaerobic THC MPN	MI SOP Anaerobic THC MPN
Media Preparation	MI SOP Media Preparation
Petroleum Degrading MPN	MI SOP Petroleum Degrading MPN
Petroleum degrading plate	MI SOP Petroleum degrading plate
Sulfate-Reducing Bacteria MPN	MI SOP SRB MPN
Total Heterotrophic Plate Count	MI SOP THC
Total Heterotrophic Count Aerobic MPN	MI SOP THC Aerobic MPN
Total Heterotrophic Count Petifilm	MI SOP THC Petrifilm

DNA:

Agarose	MI SOP agarose
Clean-up	MI SOP Clean up
DGGE	MI SOP DGGE
Extraction	MI SOP ext
Extraction-long	MI SOP ext-long
PCR	MI SOP PCR
qPCR	MI SOP qPCR
RNA Extraction	MI SOP RNA ext
	MI SOP Exporting Results from the ABI 7300 Instrument
	MI SOP Calculating QPCR Results
	MI SOP 7000ABI
	MI SOP 7000ABI Exporting Results
	MI SOP 7300ABI
	MI SOP Dilution Series
	MI SOP qPCR LOD and LOQ
	MI SOP DGGE NC
	MI SOP ext NC
	MI SOP ext-long
	MI SOP qPCR NC

Equipment:

Refrigerator/Freezer	MI SOP Refrigerator Freezer
Thermometer	MI SOP Thermometer
Centrifuge Maintenance	MI SOP Centrifuge Maintenance
Multichannel Pipette	MI SOP Multichannel Pipette
Pipette Technique	MI SOP Adjustable Pipette
Water Bath	MI SOP Water Bath

Pipette Technique
Pipette Technique
Incubators/Ovens

MI SOP Transfer Pipette
MI SOP Electric Pipette
MI SOP Incubators/Ovens

PLFA

PLFA Aqueous
PLFA Meth
PLFA SLVK
PLFA Soil
PLFA TL-Frac
PLFA Biotrap
PLFA Data Interpretation
PLFA Structural Group Reference

MI SOP PLFA Aqueous NEW
MI SOP PLFA Meth NEW
MI SOP PLFA SLVK NEW
MI SOP PLFA Soil NEW
MI SOP PLFA TL-Frac NEW
MI SOP PLFA Biotrap
MI SOP PLFA Data Interp
MI SOP PLFA Struc Grp Ref

Version	Effective	2007 Review	2008 Review	2009 Review
6/14/2006	6/14/2006	7/12/2007	5/15/2008	5/22/2009
6/15/2006	6/15/2006	7/12/2007	5/15/2008	5/22/2009
6/14/2006	6/14/2006	7/12/2007	5/15/2008	5/22/2009
6/14/2006	6/14/2006	7/12/2007	5/15/2008	5/22/2009
3/8/2008	3/10/2008	7/12/2007	5/15/2008	5/22/2009
6/21/2006	6/21/2006	7/12/2007	5/15/2008	5/22/2009
6/21/2006	6/21/2006	7/12/2007	5/15/2008	5/22/2009
6/21/2006	6/21/2006	7/12/2007	5/15/2008	5/22/2009
6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009
6/21/2006	6/21/2006	7/12/2007	5/15/2008	5/22/2009
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6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009
6/21/2006	6/21/2006	7/12/2007	5/15/2008	5/22/2009
1/6/2006	1/6/2006	7/12/2007	5/15/2008	5/22/2009
1/10/2006	1/10/2006	7/12/2007	5/15/2008	5/22/2009
4/26/2006	4/26/2006	7/12/2007	5/15/2008	5/22/2009
1/6/2006	1/6/2006	7/12/2007	5/15/2008	5/22/2009
2/27/2006	2/27/2006	7/12/2007	5/15/2008	5/22/2009
4/4/2006	4/4/2006	7/12/2007	5/15/2008	5/22/2009
1/10/2006	1/10/2006	7/12/2007	5/15/2008	5/22/2009
6/10/2006	8/10/2006	7/12/2007	5/15/2008	5/22/2009
8/10/2006	8/15/2006	7/12/2007	5/15/2008	5/22/2009
8/14/2006	8/14/2006	7/12/2007	5/15/2008	5/22/2009
6/2/2006	6/2/2006	7/12/2007	5/15/2008	5/22/2009
7/25/2006	7/25/2006	7/12/2007	5/15/2008	5/22/2009
6/2/2006	6/2/2006	7/12/2007	5/15/2008	5/22/2009
8/8/2006	8/8/2006	7/12/2007	5/15/2008	5/22/2009
8/9/2006	8/9/2006	7/12/2007	5/15/2008	5/22/2009
4/26/2006	4/26/2006	7/12/2007	5/15/2008	5/22/2009
1/6/2006	1/6/2006	7/12/2007	5/15/2008	5/22/2009
2/27/2006	2/27/2006	7/12/2007	5/15/2008	5/22/2009
1/10/2006	1/10/2006	7/12/2007	5/15/2008	5/22/2009
6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009
6/16/2006	6/16/2006	7/12/2007	5/15/2008	5/22/2009
6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009
6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009
6/16/2006	6/16/2006	7/12/2007	5/15/2008	5/22/2009
6/15/2006	6/15/2006	7/12/2007	5/15/2008	5/22/2009

6/15/2006	6/15/2006	7/12/2007	5/15/2008	5/22/2009
6/16/2006	6/16/2006	7/12/2007	5/15/2008	5/22/2009
6/22/2006	6/22/2006	7/12/2007	5/15/2008	5/22/2009

5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
5/25/2006	5/25/2006	7/12/2007	5/15/2008	5/22/2009
7/1/2006	7/5/2006	7/12/2007	5/15/2008	5/22/2009
6/26/2006	6/26/2006	7/12/2007	5/15/2008	5/22/2009

GULF COAST ANALYTICAL LABORATORIES, INC.
EXTRACTIONS
STANDARD OPERATING PROCEDUREPROCEDURES: EXT-033
PAGE: 1 of 3
EFFECTIVE DATE: 05/19/09
APPROVED BY: MAP
QA/QC APPROVED: JBT

SUBJECT SCOPE AND APPLICATION

The pH of water samples is determined electrometrically using a combination electrode. The meter is calibrated using a series of standard solutions of known pH.

MATRIX Water

REFERENCES Standard methods 4500-H⁺B Electrometric method
SW 846 9040B

PRESERVATIVE None

HOLDING TIME Analyze Immediately - Analysis should be performed as soon as possible after receipt

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

REAGENTS Deionized Water (DI)
Calibration Buffers
Buffer 1.0, 4.0, 5.0, 7.0, 8.0, 10.0, 13.0
Each aliquot of the buffers may only be used once

APPARATUS Orion 720A pH meter
Combination electrode
Temperature probe
Magnetic stir plate
Magnetic stirring bars
Plastic cups

PROCEDURE CALIBRATION

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1. Press [calibrate] button.
2. Meter will ask, how many buffers? Press [3][yes]. Each buffer should be poured into a plastic cup containing a magnetic stir bar.
3. Place electrode and temperature probe in the buffer (pH 4.00); stir gently and allow meter to stabilize; press [4.00] [yes].
4. Place electrode and temperature probe in the buffer (pH 7.00); stir gently and allow meter to stabilize; press [7.00] [yes].
5. Place electrode and temperature probe in the buffer (pH 10.00); allow meter to stabilize; press [10.00] [yes].
6. Record slope percent in calibration logbook. It will be displayed after the 3rd buffer. Slope should fall within the laboratory control limits of 92 - 108. If the slope is outside of this range, recalibrate.
7. Analyze the QC check buffer (5.00) immediately after calibration. Pour the QC check buffer into a plastic cup containing a stir bar. Place the electrode and temperature probe in the solution, stir gently and allow to stabilize. The reading must be within 0.05 pH units of the true value. If not, the meter must be recalibrated. Record result in the logbook. A QC sample must be analyzed with every 20 samples.
8. Follow the same procedure to calibrate with a different range of buffers.
9. When the probe is not in use, leave soaking in a pH 7 buffer solution.

SAMPLE ANALYSIS

1. Shake samples thoroughly, pour approximately 25-50 mL into a plastic cup containing a magnetic stir bar. Sufficient sample must be available to cover the sensing element of the electrode and to give adequate clearance for the stir bar.
2. Immerse the electrode and temperature probe in the sample. Gently stir at a constant rate to provide homogeneity and suspension of solids. Allow to stabilize.
3. Read results to the second decimal place.
4. Repeat steps 1-3 on successive aliquots of each sample until values differ by less than 0.1 pH units.
5. Thoroughly rinse electrode and temperature probe with DI water.
6. Repeat steps 1-5 for each sample. Record the first sample as the batch duplicate for each day.
7. When a sample does not fall within the current calibration range, the meter must be re-calibrated in a range to bracket the sample(s). The meter will only store one slope at a time, therefore only the last calibration is valid. Multiple calibrations may be necessary each day.

DETECTION LIMIT Lower 2.00
Upper 13.0

MAINTENANCE

1. Flush and refill electrode as needed.
2. When dirt or oil builds up on the electrode, clean with methanol.

METHOD PERFORMANCE Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as: Bias %	Accuracy as: Bias pH Units
3.5	0.10	-0.29	-0.01
3.5	0.11	-0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	-0.03	-0.002
8.0	0.13	-0.12	-0.01
8.0	0.12	+0.16	+0.01

POLLUTION PREVENTION See QAPP Section 13.2

WASTE MANAGEMENT See SOP GEN-009

QC Limits: True Value \pm 0.05 pH units
Slope Limits: 92-108

Analyst/Date: _____

Calibration Time	Buffer	Lot #	Expiration Date	Calibration Levels			Slope	QC Check	
				1	2	3		Actual	Found
1									
2									
3									
4									
5									
6									

pH RESULTS

HBN #: _____

Client	Sample ID	Time	Analyst	Result 1	Result 2	Result 3
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

Result 1 - Aliquot 1 (Repeat with successive aliquots of each sample until results agree within 0.1 pH units)

Result 2 - Aliquot 2

Result 3 - Aliquot 3

QC Check Lot# _____

Exp. _____

QC Check Lot# _____

Exp. _____

Revi

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GULF COAST ANALYTICAL LABORATORIES, INC.
EXTRACTIONS
STANDARD OPERATING PROCEDURE

PROCEDURE: EXT-026
PAGE: 1 OF 5
EFFECTIVE DATE: 05/19/09
APPROVED BY:
QA/QC APPROVED:

MAP
JDT

SUBJECT SCOPE AND APPLICATION

This Standard Operating Procedure describes method 1311 which is designed to determine the mobility of both organic and inorganic analytes present in samples of liquid, solids and multiphase wastes. This procedure is not applicable to volatile analytes.

PURPOSE For liquid samples (i.e., those containing <0.5% dry solid material), the sample, after filtration through a 0.6 to 0.8 μ m glass fiber filter is defined as the extract. For samples containing >0.5% dry solids, the liquid is separated from the solid phase and stored for later analysis. The solid phase is then extracted with a specific amount of the appropriate extraction fluid. After the appropriate extraction time, the liquid is filtered off and recombined, if compatible, with the initial liquid portion of the sample collected. If there was no initial liquid phase, the filtrate obtained is defined as the extract. In the event these two are not compatible, then they must be analyzed separately and the proper layer percentages assigned to each fraction.

MATRIX Water and Solid

REFERENCE SW846 1311

PRESERVATIVE Cool to 4°C, if Volatiles are to be extracted.

HOLDING TIME From field collection to TCLP extraction:
Volatiles - 14 days
Semivolatiles - 14 days
Mercury - 28 days
Metals - 180 days

From TCLP extraction to Preparative extraction:
Semivolatiles - 7 days
Not applicable for other analytes

From Preparative extraction to Determinative analysis:
Volatiles - 14 days
Semivolatiles - 40 days
Mercury - 28 days
Metals - 180 days

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle

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labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

APPARATUS

Agitation apparatus capable of end-over-end rotation @ 30 ± 2 rpm
Plastic extraction bottles (metals only) - TCLP jug
Teflon extraction bottles - TCLP jug
Filter holder - 150mm polypropylene
Glass fiber filters, 150mm, 0.6 to 0.8 μ m nominal pore size
pH meter (accurate to 0.01 pH unit)
pH electrode with automatic temperature compensation
Analytical balance - accurate to 0.01 grams
Spatula
4oz Wide-mouth jar
Hot plate/ magnetic stirrer
Magnetic stir bars
Graduated cylinders - 1000 mL glass
Vacuum manifold and pump
1 mm and 9.5 mm sieves
1 L Plastic bottles (metals extract)
1 L Amber glass bottles (organics extract)

REAGENTS

All reagents shall be reagent grade or equivalent. Label all containers and squeeze bottles with reagent ID, lot, and expiration date.

Deionized water
Glacial acetic acid
Sodium hydroxide 50%
Hydrochloric acid (1N) - 8.3 mL HCl to 100 mL DI water
1:1 Nitric acid
pH Buffers (calibration)

EXTRACTION FLUID #1

5 gallons DI water
114 mL Acetic acid
70 mL 50% NaOH
pH = 4.93 ± 0.05 pH units (4.88-4.98)

EXTRACTION FLUID #2

5 gallons DI water
114 mL Acetic acid
pH = 2.88 ± 0.05 pH units (2.83-2.93)
Record in the preparation logbook and assign a laboratory ID.

PROCEDURE

1. Determination of percent solids:
 - A. Liquid wastes - wastes with <0.5% solids after filtration through a 0.6 to 0.8 μ m glass fiber filter is defined as the TCLP extract.
 - B. 100% solids - samples that will yield no liquid when subjected to pressure filtration. Homogenize the sample by pouring the contents of the sample container onto a piece of butcher paper, chop and mix with a tongue

depressor or spatula. Remove any foreign object such as sticks, leaves, or rocks. If the particle size is > 9.5 mm, perform particle size reduction by crushing, grinding, or cutting the sample. The sample may be sieved to verify proper particle size.

C. Multiphasic - a determination of the % solids must be performed as outlined below:

1. Weigh a 0.8 um filter and place on screen of the filter apparatus. Assemble the filter apparatus.
2. Weigh a wide mouth quart jar.
3. Weigh 100 grams of sample - record weight in logbook. Allow to settle or centrifuge.
4. Place jar under filter apparatus.
5. Pour liquid sample into the filtering apparatus followed by the solid portion of the sample. If there is obvious residue left in the cup, reweigh the cup and subtract the weight of the residue from the weight of the sample to be filtered.
6. Pressurize Millipore filter apparatus to 10 psi and let sit for 2 minutes to a maximum of 50 psi.
7. Relieve pressure from the filter apparatus when the gas flows through the filter or when there is more than 2 minutes between drops of filtrate.
8. Weigh the jar and filtrate. Subtract the weight of the jar from the jar and filtrate weight. This is the liquid phase.
9. Subtract the liquid phase from the weight of the total sample. This is the solid phase.

$$\% \text{ Solids} = \frac{\text{weight of solid phase}}{\text{total weight of sample}} \times 100$$

If the % solids is >0.5%, determine if the solid needs particle size reduction. Follow the procedure in B to perform particle size reduction. If it is noticed that filtrate may be entrained in the filter then remove the solid phase and filter and dry at $100 \pm 20^\circ\text{C}$ cycles until two successive weightings are, within $\pm 1\%$ of each other. Calculate the % dry solids. If the % dry solids are <0.5%, then the filtrate is the TCLP extract.

D. Determination of extraction fluid
Solids from samples which are 100% solid or >0.5% solids (solid portion) are prepared and extracted as follows.

1. Label with the sample number, a 4 oz. Wide-mouth jar and TCLP jug. A plastic TCLP jug may be used if the extract is to be analyzed for metals only. If organics will be analyzed, a Teflon TCLP jug must be used.
2. Weigh a small sub-sample of the solid phase of the waste and reduce to a particle size of approximately 1 mm. Sieve if necessary. Transfer

5.0 grams of sample into the 4 oz Wide-mouth jar.

3. Add 96.5 mL (\pm 1mL) of DI water to the 4 oz. Wide-mouth jar. Add a stir bar, and place the sample on a magnetic stirrer and stir vigorously for 5 minutes. Measure and record the pH in the TCLP log book. If the pH is <5 , extraction fluid #1 shall be used. If the pH measured is >5 , add 3.5 mL of 1.0 N HCL to the jar and slurry the mixture. Heat to 50°C . Hold at this temperature for 10 minutes. Remove from the hot plate and measure the pH. If the measured pH is <5 , then extraction fluid #1 will be used. If it is >5 , then extraction fluid #2 must be used, Record the fluid used in the logbook.

E. Extraction:

1. Weigh 100g of 100% solids into the labeled TCLP jug. Weigh 100g of % solid samples, filter, and place the filter and solids into the labeled TCLP jug. Store the liquid phase at 4°C . If there is not 100g of the solid or %solid sample, contact client services immediately and wait for further instructions. Do not perform TCLP extraction on a smaller sample aliquot without authorization from the client.
2. Slowly add the appropriate extraction fluid in the amount of 20 times the sample weight (accounting for % solids).
3. Secure the bottle in the agitation device and rotate at 30 rpm for 18 ± 2 hours. The temperature must be maintained at $23 \pm 2^{\circ}\text{C}$ during the TCLP extraction. Document the max and min temperature in the TCLP logbook.
4. After rotation, allow the sample to stand and settle for approximately 30 minutes before filtration.
5. Assemble a filtration vessel and filter the sample through a new acid rinsed filter paper. Vacuum filtration can only be used for wastes with $<10\%$ solids content.
6. After the sample has been filtered, for 100% solids, the filtrate is the TCLP extract. For % solid samples, combine the filtrate with the initial liquid phase if compatible. If incompatible, each phase is prepped and analyzed separately.
7. Record the pH. Preserve the metals aliquot of the extract to a pH of <2 with 1:1 Nitric acid.
8. Store extracts at 4°C until ready to proceed with the preparation and/or analysis procedures required for the analytes of interest.
9. Notify metals prep analyst of TCLP extracted samples needing metals analysis. Extract samples

for semivolatile analysis using applicable SOP.

METHOD PERFORMANCE

Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP Toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

- 1) *Metals* - The results of a multi-laboratory study are shown in Table 6 of Method 1311, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.
- 2) *Semivolatile Organic Compounds* - The results of two studies are shown in Tables 7 and 8 of Method 1311. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20-120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

POLLUTION PREVENTION

See QAPP Section 13.2

WASTE MANAGEMENT

See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
WET LAB
STANDARD OPERATING PROCEDUREPROCEDURE: WL-063
PAGE: 1 of 5
EFFECTIVE DATE: 02/13/09
APPROVED BY: *MAP*
QA/QC APPROVED: *JBT*

SUBJECT SCOPE AND APPLICATION

Alkalinity of water is its acid-neutralizing capacity. It is the sum of all the titrable bases. An unpreserved sample is titrated electrometrically to a fixed pH end point using a Mettler-Toledo DL53 autotitrator.

MATRIX Water

REFERENCE Standard Methods, 2320B 18th Edition

PRESERVATIVE Cool 4°C

HOLDING TIME 14 days

SAMPLE COLLECTION Samples should be collected in Polyethylene or glass bottles. Sample containers should be completely filled and capped tightly. Avoid sample agitation and prolonged exposure to air.

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents and chemicals must be handled in ventilated hoods.

REAGENTS 0.02N Sulfuric Acid (H₂SO₄), Commercially prepared
0.02N Sodium Hydroxide (NaOH), Commercially prepared.
pH Buffers 4.00, 7.00, 10.00, Commercially prepared.
Deionized WaterAPPARATUS Mettler Toledo DL53 Autotitrator
DG111-SC Combination pH Electrode
100 mL Plastic cups
10 mL Burette
5 & 10 mL Disposable Pipettes
10mL class "A" volumetric pipette

100 mL Graduated Cylinder
Printer

INTEFERENCES

Soap, oil matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response.

PH CALIBRATION

- 1) Press the green run button on DL53.
- 2) In the number of samples line, type in the number 3.
- 3) In the method ID line, type in the number 1 and press enter on the keyboard.
- 4) Pour the contents of a pH buffer 4.00 pack into a 100 mL plastic cup and load onto the DL53 Autotitrator. Press enter on the keyboard.
- 5) Pour the contents of a pH buffer 7.00 pack into a 100 mL plastic cup and load onto the DL53 Autotitrator. Press enter on the keyboard.
- 6) Pour the contents of a pH buffer 10.00 pack into a 100 mL plastic cup and load onto the DL53 Autotitrator. Press enter on the keyboard.
- 7) Upon completion of the 3rd buffer, a printout should appear on the printer. On the printout is the slope. The slope should read between -52 and -65 in order for the calibration to be accepted.
- 8) Press F5 on the DL53.

QUALITY CONTROL

- 1) Check titrant bottle on DL53.
- 2) Make sure the correct burette is installed on DL53.
- 3) To prepare an LCS 200 mg/L use a 10 mL class "A" volumetric pipette to measure 10 mL of 0.02N NaOH into a 50 mL volumetric flask and diluting to a total volume with deionized water. Once prepared, transfer LCS to a 100 mL plastic cup.
- 4) In the method ID line, type in the number 12 and press enter on the keyboard.
- 5) Scroll up to the sample ID line and type in "LCS200mg/L".
- 6) Scroll down to the volume line and type in "50" and press enter on the keyboard.
- 7) Load 100 mL plastic cup on the DL53 Autotitrator and press enter on the keyboard again.
- 8) Upon completion, a report will print. The LCS should read 200 mg/L \pm 10% in order for analysis to continue.

- 9) Press F5 on DL53.

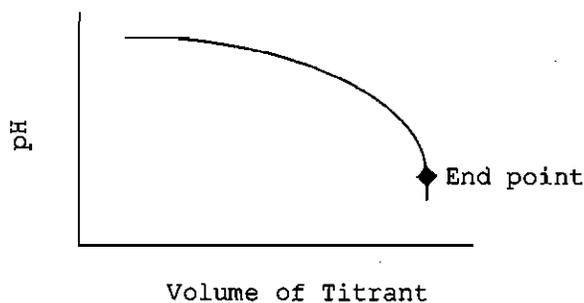
PROCEDURE

- 1) Allow samples to warm to room temperature before analysis.
2) Pour up a 50 mL aliquot of the sample to be analyzed into a 100 mL plastic cup.

ALKALINITIES REQUESTED	METHOD ID
ALKT Only	12
ALKP Only	13
Both ALKT and ALKP	11
ALKB, ALKC, or ALKOH	11

- 3) Using the above chart, type in the correct method ID for the sample of interest.
4) Press enter on the keyboard.
5) Scroll up to the sample ID line and type in the appropriate sample number or sample ID.
6) Scroll down to the volume line and type in "50" and press enter on the keyboard.
7) Press enter one last time.
8) Upon completion of analysis, a report will print showing the results. The results printout will include a titration curve of volume of titrant versus pH. As the pH approaches the endpoint of 4.5, the curve should quickly drop off to an inflection point. An example of a curve is below.

Example: Titration Curve



- 9) Press F5 on the DL53.
10) Follow steps 1-9 for any additional samples.

NOTES:

- 1) Any sample which has a total alkalinity of <20 mg/L should be rerun according to the Low Alkalinity Standard Operating Procedure (WL-064).
- 2) Up to 20 samples may be batched together and each batch should contain a LCS and a duplicate. The duplicates should agree within 10% RPD.
- 3) When analyzing for ALKP, if the initial pH of the sample is below pH 8.3, then the sample should be reported as <1.0 mg/L CaCO₃.
- 4) Samples that reach the maximum titrant volume of 20 mL should be reanalyzed using a smaller aliquot of sample.

CORRECTIVE ACTION

- 1) If the LCS falls outside of acceptance limits of 90-110%, the analysis should be stopped immediately. Any samples that have been analyzed must be rejected and must be repeated.
- 2) If duplicate analysis falls outside of the 10% RPD limit, duplicate analysis may be repeated one time. If the reanalyses still yields results outside of the RPD criteria, all samples in the batch should be reanalyzed.

CALCULATIONS

All calculations are performed by the DL53 autotitrator software if the information for each sample is entered correctly. The following calculations are the ones used by the instrument.

$$\text{ALKT, mg/L CaCO}_3 = \frac{(\text{mL of titrant to pH 4.5})(0.02\text{N})(50,000)}{\text{mL of sample}}$$

$$\text{ALKP, mg/L CaCO}_3 = \frac{(\text{mL of titrant to pH 8.3})(0.02\text{N})(50,000)}{\text{mL of sample}}$$

Hydroxide (ALKOH), Carbonate (ALKC), and Bicarbonate (ALKB) alkalinities are determined based on the relationship of the phenolphthalein and total alkalinities. These values are not calculated by the instrument, but should be calculated by the analyst using the following chart. All results are reported as mg/L CaCO₃.

<u>Result of Titration</u>	<u>Hydroxide Alkalinity</u>	<u>Carbonate Alkalinity</u>	<u>Bicarbonate Alkalinity</u>
P = 0	<1.0	<1.0	T
P < ½ T	<1.0	2P	T - 2P
P = ½ T	<1.0	2P	<1.0
P > ½ T	2P - T	2(T - P)	<1.0
P = T	T	<1.0	<1.0

T = Total Alkalinity

P = Phenolphthalein

BURETTE CALIBRATION - The burette drive used on the DL53 Autotitrator yields 5000 to 1 accuracy to volume ratio. For example, using the 10mL burette results in 0.002 mL burette accuracy. The DL53 Autotitrator burette is calibrated annually by Mettler Toledo during scheduled annual preventive maintenance. Approximately 1 month before the maintenance is required, contact Mettler Toledo to schedule the annual preventive maintenance and burette calibration. Once the preventive maintenance and calibration have occurred, the event should be documented in the maintenance logbook for the DL53 Autotitrator.

DETECTION LIMIT 20.0 mg/L CaCO₃

METHOD PERFORMANCE Seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/liter, CaCO ₃	Precision as Standard Deviation mg/liter, CaCO ₃	Accuracy as Bias, %	Accuracy as Bias mg/l CaCO ₃
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

POLLUTION PREVENTION See QAPP Section 13.2

WASTE MANAGEMENT See SOP GEN-009

GULF COAST ANALYTICAL LABORATORIES, INC.
WET LAB
STANDARD OPERATING PROCEDURE

PROCEDURES: WL-021
PAGE: 1 OF 7
EFFECTIVE DATE: 05/19/09
APPROVED BY: MAP
QA/QC APPROVED: JDT

SUBJECT SCOPE AND APPLICATION

This is a measure of the organic matter content in a sample that can be oxidized by a strong chemical oxidant. This method must be performed by or under the supervision of a trained analyst. See SOP QA-014 for demonstration of capability requirements.

MATRIX Water

REFERENCES HACH Method 8000 (Reactor digestion method)
COD Control Software Manual

PRESERVATION Sulfuric Acid, stored at 4°C

HOLDING TIME 28 Days

DEFINITIONS See SOP GEN-016

SAFETY Use normal safety laboratory procedures. Gloves safety glasses and protective clothing are required.

INTERFERENCES Chloride is the primary interference when determining COD. Each COD vial contains Mercuric sulfate that will eliminate Chloride interference up to 2000 mg/L. Samples with higher concentrations of Chloride must be diluted. The interference is often noticed if the reagents turn green after addition of the sample to the vial.

APPARATUS

Disposable plastic pipets
Hach reactor digestion block
Volumetric flasks 100ml
Specimen cups
Plastic cups
Kim-wipes
Timer
Thermometer for digestion block
Test tube rack
Mechanical pipet and tips - accuracy 2%
HACH DR2800 Spectrophotometer & vial adapter
Calculator
Fine-tip marker

A Maintenance logbook is issued for each instrument. All maintenance performed by the analyst, GCAL employee, or service representative must be entered into the logbook.

STANDARDS AND
REAGENTS

All standards used must pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Follow manufacturer's instruction for standard expiration and storage. All standards must have the date opened and expiration date. COD vials are purchased from HACH and contain all necessary reagents. Store as instructed.

Calibration Standard - COD 1000 mg/L
commercially prepared

Secondary Source Standard (LCS)- COD 1000 mg/L,
commercially prepared

Deionized water (DI)

Low range COD vials, 0 - 150 mg/L

PROCEDURE

CALIBRATION

1. Recall the user program for COD.
2. Set wavelength to 420 nm. Preheat the reactor digestion block for about ½ hour before you start. The temperature is maintained at $150^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
3. Prepare a calibration curve or verify an existing curve.

NOTE: Analysts performing analysis on North Carolina samples must prepare their own standard curve.

4. To prepare a calibration curve, prepare the following standards. The calibration standards are prepared from the 1000 mg/L stock standard. The standards are diluted with deionized water.

5.0 mg/L -- Add 0.5 mL of the 1000 mg/L standard to volumetric flask and fill to 100 mL.

25 mg/L -- Add 2.5 mL of the 1000 mg/L standard to a volumetric flask and fill to 100 mL.

75 mg/L -- Add 7.5 mL of the 1000 mg/L standard to a

volumetric flask and fill to 100 mL.

100 mg/L -- Add 10.0 ml of the 1000 mg/L standard to a volumetric flask and fill to 100 mL.

150 mg/L -- Add 15.0 ml of the 1000 mg/L standard to a volumetric flask and fill to 50 mL.

The blank for the curve is deionized water.

5. Using a mechanical pipet measure 2 mL of the blank and each standard and place in a COD vial. The CCV is prepared by Pipetting 1.85 mLs of DI water and 0.15 mLs of calibration standard. Repeat using second source standard for the LCS. Using a fine-tip marker, label the vial by writing on the cap of the vial. Be sure to tip the vial and the pipette at a 45-degree angle when placing the sample in the vial. Put cap on and gently shake the vial over the sink. Vial will get very hot. Place vial in digestion block. (Note: mechanical pipettes must be calibrated each day before use, see SOP GEN-005.)
6. Cook the vials for two hours at 150°C. Record the start and stop time and the temperature on the HACH Standard ID worksheet.
7. Remove the vials from the digestion block and invert several times gently. Place in cooling rack for a minimum of 30 minutes.
8. Read the vials on the Spectrophotometer set at 420 nm. Read the vials by placing in the holder with the vial centered between the A & C in HACH positioning the vial with the HACH logo facing the front of the instrument. This insures consistent readings. Wipe the vial with a clean Kim-wipe before placing in the holder. Close the HACH light shield.
9. Perform the following steps to calibrate the HACH:
 - a. Select "USER PROGRAM"
 - b. Select method (test) to calibrate.
 - c. Select "EDIT", press enter.
 - d. Select "Calib.table", press "EDIT TABLE".
 - e. Select "EDIT ABS".
 - f. Press "CE" on each line until all ABS are clear.
 - g. Select "ENTRY DONE".
 - h. Zero with a blank and begin measurement of standards.

- i. Select "ENTRY DONE".
 - j. Select "EXIT".
 - k. Select "yes" to save changes.
 - l. Edit calibration table.
 - m. Print graph and print full table
 - n. Run ICV/LCS $\pm 10\%$ and ICB $< \frac{1}{2}$ RL & LLC - low level standard $\pm 50\%$.
10. An existing curve can be verified by analyzing the 75 mg/L working standard (CCV) processed through steps 5-8. The recovery must be 90-110% to continue the analytical run. A method blank must also be analyzed.
 11. A new curve must be performed when the CCV acceptance criterion is not met, the standard expires, or at least annually.

SAMPLES

1. Create a batch in the LIMS. Print the batch sample worklist (BSWA). Use the BSWA and barcode reader to enter the sample IDs as the samples are analyzed.
2. Write the sample ID on the vial cap.
3. Shake samples to homogenize. If sample has a large amount of visible solids, allow to settle, and shake to homogenize. Using a mechanical pipet place 2 mL of sample in a COD vial. If solution turns green, the COD is very high or Chloride interference is present. Dilute the sample before adding to the vial. Be sure to tip the vial and the pipette at a 45-degree angle when placing the sample in the vial. Write the dilution on the vial. Put cap on and gently shake the vial over the sink. Vial will get very hot. Place vial in digestion block.
4. Proceed with steps 2 and 6-8 of the calibration procedure. Dilute and reanalyze samples that exceed the upper calibration range of the instrument.
5. After batch analysis is completed, save the data on the HACH by the HBN number on "BSWA". Upload data to the LIMS. Record all required information on cover page and attach to the printout of the raw data.

6. Calculate % recovery and record on raw data.

QUALITY CONTROL SAMPLES

1. Analyze an ICV (2 mL of the second source standard), the low level standard, and the blank immediately after an initial calibration. The ICV recovery must be 90-110%. If not prepare, a new ICV and reanalyze. If the recovery is still outside the control limits a new calibration must be performed. The recovery for the low level standard must be 50-150%. If the recovery is outside these limits, a new calibration must be performed.
2. Analyze one method blank with every 20 samples. The blank is deionized water processed as a sample. If the reading is above this value, prepare a new blank and rerun. If the value is still above this value, prepare a new calibration. The concentration of the method blank must be less than $\frac{1}{2}$ the reporting limit, less than 5 % of the associated samples, or be associated with non-detect concentrations in the samples. The sample batch or affected samples must be reanalyzed if the method blank criteria are not met.
3. Analyze one LCS with every 20 samples. Use 2 mL of the independent working standard processed as a sample. A LCSD is performed if insufficient sample is available to perform a duplicate or if required for a specific project. The LCS recovery must be 80-120%. If the LCS recovery is high and associated samples are non-detect, the data may be reported with a narrative. All other failures of the LCS require reanalysis of the sample batch. A LCSD is performed if insufficient sample is available to perform a batch duplicate. The LCS/LCSD RPD must be ≤ 25 .
4. Duplicate one of every twenty samples. Process a second aliquot of the chosen sample. The duplicate RPD must be ≤ 25 for concentrations \geq five times the reporting limit. If the RPD is above the control limit, reanalyze the sample batch if the exceedance is not caused by a non-homogeneous sample.
5. Analyze one matrix spike for every twenty samples. Add

0.15 mL of the 1000 mg/L LCS/ICV standard and 1.85 mL of the sample chosen for spiking to the vial. The matrix spike recovery is 75-125%. If the recovery is outside the control limits, report the data with a narrative or reanalyze to verify the presence or absence of matrix interference. It may be necessary to reanalyze the batch spike sample diluted to eliminate interference. A MSD is performed is requested by the client or required in a project plan. The MS/MSD RPD is ≤ 25 .

6. Analyze a CCV/CCB every 10 samples. The CCV is 0.15 mL of the standard and 1.85 mLs of DI water and the CCB is 2 mL of deionized water. The CCV recovery must be within 90-110%. If the recovery for the CCV is outside the acceptance limits, rerun the CCV. If the recovery is now acceptable, report the data. If an acceptable CCV cannot be analyzed, determine the problem and recalibrate the instrument if necessary. Any samples that are bracketed by failing CCVs must be reanalyzed. Non-detect values may be reported with high recovery CCVs with a narrative. The CCB concentration must meet the same criteria as the method blank.
7. Projects may have more stringent criteria that must be met for the associated samples. See SOP GEN-019.

NOTE: COD may have a correlation with TOC & BOD for some samples. COD should be 2 to 5 times greater than TOC. COD is > BOD.

CALCULATIONS

mg/L O₂ = Instrument reading * Dilution Factor (this must be less than or equal to 99999 or the LIMS will not report the correct value)

Percent Recovery = (C/T)*100 where:
C is the determined concentration and
T is the theoretical concentration

Precision as RPD = $\frac{|D_1 - D_2|}{\frac{(D_1 + D_2)}{2}} \times 100$ where

D₁ is the concentration of the first determination and
D₂ is the concentration of the duplicate

All calculations performed by the software including curve fitting and correlation coefficient are listed in the COD

Control Software Manual, page 165.

REPORTING
LIMIT

5.0 mg/L

METHOD
PERFORMANCE

In a single laboratory using standard solutions of 100 mg/L COD and 500 mg/L COD and two lots of reagent with the DR/2000, a single operator obtained a standard deviation of, ± 2.7 mg/L COD for the 0 to 150 mg/L range vials.

POLLUTION
PREVENTION

See QAPP Section 10.2

WASTE
MANAGEMENT

See SOP GEN-009

HACH STANDARD IDS

Analyst _____
Date _____
HBN# _____
Batch# _____

SULFIDE 4500 S D/HACH 8131
CCV Lot# _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
Sulfide Reagent 1 Lot# _____
Sulfide Reagent 2 Lot# _____
Calibration Date _____
Calibration Expiration Date _____

HEXAVALENT CHROMIUM 7196A/3500 Cr B-01
CCV Lot# _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
Powder Pillow Lot # _____
Calibration Date _____
Calibration Expiration Date _____

SULFATE 300.0/9038
CCV Lot # _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
Powder Pillow Lot# _____
Conditioning Reagent Lot# _____
Barium Chloride Lot # _____
Calibration Date _____
Calibration Expiration Date _____

ORTHOPHOSPHATE 365.1
CCV Lot# _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
Powder Pillow Lot # _____
Calibration Date _____
Calibration Expiration Date _____

FLUORIDE 340.1
CCV Lot # _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
SPADS Lot# _____
Calibration Date _____
Calibration Expiration Date _____

CHLORINE (Total Residual/Free) 4500-L G/4500-CL D
CCV Lot# _____
CCV Expiration Date _____
ICV/LCS Lot # _____
ICV Expiration Date _____
Powder Pillow Lot # _____
Calibration Date _____
Calibration Expiration Date _____

COD HACH 8000
COD Reactor # _____
CCV Lot # _____
CCV Expiration Date _____
ICV/LCS Lot# _____
ICV Expiration Date _____
COD Vials Lot# _____
Calibration Date _____
Calibration Expiration Date _____
Digestion Start/Stop Time _____
Digestion Temperature (°C) _____

Ferrous Iron 3500-Fe D
CCV Expiration Date _____
ICV/LCS Lot# _____
ICV Expiration Date _____
Ferrous Ammonia Acetate Lot# _____
Phenanthroline Lot# _____
Calibration Date _____
Calibration Expiration Date _____
CCV Lot# _____

REVIEWED BY: _____

Rev

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GULF COAST ANALYTICAL LABORATORIES, INC
EXTRACTIONS
STANDARD OPERATING PROCEDURES

PROCEDURE: EXT-003
PAGE: 1 of 5
EFFECTIVE DATE: 07/07/08
APPROVED BY: SKB
QA/QC APPROVED: JAT

SUBJECT SCOPE AND APPLICATION

This procedure is designed for the preparation of aqueous samples and TCLP extracts which may contain Extractable Base/Neutral/Acid organic compounds. A measured volume of sample, approximately 1.0 L, is serially extracted with methylene chloride at a pH > 11 and again at a pH < 2.

MATRIX Water

REFERENCE SW-846 Method 3510C
EPA 625

PRESERVATIVE Cool to 4°C

HOLDING TIME From collection to extraction - 7 days
From extraction to analysis - 40 days

TCLP:
From collection to TCLP extraction - 14 days
From TCLP extraction to preparative extraction - 7 days
From preparative extraction to analysis - 40 days

DEFINITIONS See SOP GEN-016

SAFETY 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. In all cases, the applicable material safety data sheet (MSDS) and your supervisor or safety officer should be consulted. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and lab coats must be worn. In addition to other measures prescribed by the division, solvents must be handled in ventilated hoods.

APPARATUS Water bath
Sample shaker
Separatory Funnel -- 2 L with Teflon stopcock
Kuderna-Danish (K-D) apparatus
A) Concentrator tube (receiver)-- 10 mL, graduated
B) Evaporation flask -- 500 mL, attach to tube with blue clamp
C) Snyder column -- three-ball macro
D) Snyder column -- two-ball micro

Glass Wool
Glass funnels - pack with glass wool and fill with sodium sulfate; rinse with methylene chloride; collect rinse in an

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Erlenmeyer flask and then discard in the appropriate waste container

Erlenmeyer flask
Boiling chips, Teflon
pH indicator paper with pH range 2-14
Graduated cylinders -- 1000 mL and 100 ml
1.0 mL pipettor
Glass beaker for micro-Snyder process
Hot plate
Glas-col mechanical shaker
1.0 mL class A volumetric flask

REAGENTS

All organic reagents used must be of pesticide quality, inorganic reagents must be reagent grade. Label prepared solutions as described in SOP GEN-006.

Deionized water
Sodium hydroxide solution, 20% (ACS) -- 320 g NaOH dissolved in 1.6 L reagent water
Sodium sulfate (ACS) -- granular, anhydrous
Sulfuric acid solution, (1:1) -- 500 ml H₂SO₄ (sp. gr. 1.84) added slowly to 500 ml reagent water
Methylene chloride

STANDARDS

All standards used must pure material or from prepared certified solutions. The certificate of analysis shall be kept on file. Label prepared standards as described in SOP GEN-006.

1. Surrogate Standard Spiking Solution

Surrogate standards are added to all samples (including QC).

Stock surrogate standard - a purchased standard in methanol containing the following surrogates at the listed concentration:

<u>50 ug/mL</u>	<u>100 ug/mL</u>
Nitrobenzene-d ₅	Phenol-d ₅
2-fluorobiphenyl	2-Fluorophenol
Terphenyl-d ₁₄	2,4,6-Tribromophenol

Store the spiking solutions at 4°C in Teflon-sealed containers. The solutions must be replaced when expired (manufacturer's date), or sooner if comparison with quality control check samples indicate a problem.

2. Spiking Solutions (8270C/625) - stored at 4°C in Teflon-sealed containers until manufacturer's expiration date.

A. 625 Spiking Solution - full list of compounds. The spiking solution is purchased at a concentration of 100 ug/mL in methanol. Spike 1.0 mL of the 625 spiking solution into the LCS and MS performed for Method 625. A MSD is not required for method 625. The spiking solution is purchased at a concentration of 100 ug/mL and is spiked directly into the LCS and MS.

B. Additional spiking solutions may be purchased to include additional analytes requested by the client.

PROCEDURE

1. Rinse all separatory funnels with methylene chloride two times. Discard solvent rinse into waste container.
2. Label each separatory funnel with the sample ID to be extracted. Complete the laboratory extraction sheet with the sample IDs. Fill in the extraction sheet information as each item is completed. An extraction sheet is attached.
3. Measure 1000 mL DI water with a graduated cylinder and pour into the separatory funnels labeled blank, LCS and LCSD.
4. With each 8270C extraction batch a method blank, LCS, LCSD and MS/MSD are included. With each 625 extraction batch a method blank, LCS, and MS are included. Document on the extraction log if insufficient sample is available for a MS and/or MSD.
5. Shake and mix sample well. Using a 1 L graduated cylinder, measure out a 1 L sample aliquot and place it into a 2 L separatory funnel. (If sample size does not permit a full 1 L volume, the volume actually extracted must be recorded in the extraction log and the analysis must reflect the true original volume of sample on the appropriate bench sheet.) In the extraction for TCLP, measure 200 mL of sample.
6. Add 1.0 mL of the surrogate standard spiking solution to each separatory funnel and mix well. Spike the LCS/LCSD and the MS/MSD with 1.0 mL of the required spiking solution.
7. Add approximately 3 mL of 1:1 sulfuric acid to each separatory funnel. Check the pH and verify it is <2. Additional acid is added if the pH is not <2.
8. Use 60 ml of methylene chloride to rinse the sample bottle and graduated cylinder (after rinsing the bottle, transfer to the graduated cylinder). (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 request both phases analyzed, separate the phases and proceed. Solids are extracted using SOP EXT-001). Transfer this rinse solvent to the separatory funnel. Shake the funnel and vent. Place the funnel on the 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 glass funnel and collect in a K-D concentrator flask, fitted with a 10 ml graduated receiver. Using the methylene chloride rinse bottle, rinse the funnel with a stream of methylene chloride and collect in the K-D concentrator.

9. Add a second 60 ml volume of methylene chloride to the 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 #8. Perform a third extraction in the same manner as the second.
10. Add approximately 20 mL of the sodium hydroxide solution to each funnel. 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 extracts as above and label as BNA.
11. Using the methylene chloride rinse bottle rinse the funnel and sodium sulfate thoroughly with a stream of methylene chloride and collect the rinse in the KD flask.
12. Rinse the Snyder column with methylene chloride. Attach the Snyder column to the evaporative flask. Place the K-D apparatus on a concentration unit. 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 5.0 - 6.0 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and evaporation flask.
13. Rinse a two-ball micro-Snyder column with approximate 0.5 mL of methylene chloride. Add another clean boiling chip to the concentrator tube and attach the two-ball micro-Snyder column. Place the 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

about 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. Adjust the final volume to 1.0 ml with methylene chloride in a 1.0 mL Class A volumetric tube. Transfer the extract to a Teflon-sealed crimp cap autosampler vial and label as BNA, as appropriate.

14. Enter the prep batch information in the LIMS. Sign the extraction sheet. Deliver a copy of the extraction log and the extracts to the GCMS Semivolatiles laboratory. If an analyst is not available to receive the samples, place in the appropriate refrigerator.
15. Extracts are analyzed using SOP GCMSSV-001 or GCMSSV-002.

METHOD
PERFORMANCE

- 1) The recovery of surrogates is used to monitor unusual matrix effects, sample processing problems, etc. The recovery of matrix spiking compounds, when compared to laboratory control sample (LCS) recoveries, indicates the presence or absence of unusual matrix effects.
- 2) The performance of each 3500 method will be dictated by the overall performance of the sample preparation in combination with the cleanup method and/or the analytical determinative method.

POLLUTION
PREVENTION

See QAPP Section 13.2

WASTE
MANAGEMENT

See SOP GEN-009

METHOD
MODIFICATIONS

The following are modifications of EPA 625 to correspond with SW-846 3510 guidance

- 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.
- 2) The base-neutral and acid extracts are combined prior to concentration.

Standard Label and Chain-of-Custody Record

Project Name: _____ Project No: _____

Sample ID: _____

Sample Date: _____ Sample Time: _____

Sampler(s): _____

Analyses: _____

Preservatives: _____

**Data Validation Checklists for SW-846 Methods,
Level IV Data Package Deliverables, and Data
Reporting Form**

DATA DELIVERABLE PACKAGE REQUIREMENTS

Method	Deliverable Requirement	Equivalent EPA Form	Preliminary Results	CCI Level A	CCI Level B	CCI Level C
Organics by GC or HPLC	Case Narrative (See Note 1)			X	X	X
	Corrective Action Report			X	X	X
	Cross-reference of CCI Sample Numbers, Lab IDs, and analytical QC batches	IV	X	X	X	X
	Chain-of-Custody Form, Cooler Receipt form		X	X	X	X
	Data Summary (Form 1) for each blank and sample (See Note 2)	I	X	X	X	X
	Blank Spike or Lab Control Sample (LCS) results (including concentration spiked, percent recovered, percent recovery acceptance limits) Note: The LCS shall be spiked with all analytes of interest.		X	X	X	X
	Surrogate Recovery Report (including concentration spiked, percent recovered, and percent recovery acceptance limits)	II	X	X	X	X
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Report (including concentration spiked, percent recovered, percent recovery acceptance limits, relative percent difference (RPD), and RPD acceptance limits) Note: MS/MSD must be performed on each batch of 20 Navy samples. Matrix spike QC must contain all the targeted analytes of interest at a concentration not less than 10 times the MDL. When using analytical methods for determination of classes of compounds (e.g. fuels analysis for TPH-gasoline or TPH-diesel), the matrix spike must contain each targeted class of compounds.	III	X	X	X	X
	Initial Calibration Data for each column (indicate which column was used for quantitation) Note: All analytes must meet calibration acceptance criteria prior to analysis of samples.	VI			X	X
	Continuing Calibration Identification Summary for Single or Multicomponent Analytes (Pest/PCBs only)	X			X	X
	Second Source Verification (including acceptance limits) Note: Result should be reported in terms of percent recovery failing within (75-125%)				X	X
	Continuing Calibration Data (indicate which column was used for quantitation) Note: All analytes must meet calibration acceptance criteria prior to analysis of samples.	VII			X	X
	Chromatograms for each sample (and reruns), confirmation runs, blank, spike, duplicate, and standards					X
	Raw Quantitation Report (area vs. retention time)					X
	Evidence of Manual Intergrations					X
	Copies of Sample Preparation Bench Sheets				X	X
	Copies of Standard Preparation Logs					X
Copies of Run Logs	VIII				X	
EDD				X	X	X

DATA DELIVERABLE PACKAGE REQUIREMENTS

Method	Deliverable Requirement	Equivalent EPA Form	Preliminary Results	CCI Level A	CCI Level B	CCI Level C
Organics by GC/MS	Case Narrative (See Note 1)			X	X	X
	Corrective Action Report			X	X	X
	Cross-reference of CCI sample numbers, Lab IDs, and analytical QC batches	IV	X		X	X
	Chain-of-Custody Form, Cooler Receipt Form		X	X	X	X
	Data Summary (Form 1) for each blank and sample (See Note 2)	I	X	X	X	X
	Tentatively Identified Compounds (TICs) for each sample (twenty peaks) – Not required unless specified in project instructions	I,TIC				
	Blank Spike/Lab Control Sample (LCS) results (including concentration spiked, percent recovered, percent recovery acceptance limits) Note: The LCS shall be spiked with all analytes of interest.		X	X	X	X
	Surrogate Recovery Report (including concentration spiked, percent recovered, and percent recovery acceptance limits)	II	X	X	X	X
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Report (including concentration spiked, percent recovered, percent recovery acceptance limits, relative percent difference (RPD), and RPD acceptance limits) Note: MS/MSD must be performed on each batch of 20 Navy samples. Matrix spike QC must contain all the targeted analytes of interest at a concentration not less than 10 times the MDL.	III	X	X	X	X
	Instrument Performance Check (Tuning) Report including raw data.	V			X	X
	Initial Calibration Data (including acceptance limits)	VI			X	X
	Second Source Verification (including acceptance limits) Note: Result should be reported in terms of per cent recovery failing within (75-125%)				X	X
	Continuing Calibration Data (including acceptance limits) Note: All analytes must meet calibration acceptance criteria prior to analysis of samples. The GC/MS calibration check solutions must include all targeted analytes, including all non-CCC and non-SPCC compounds.	VII			X	X
	Internal Standard Areas and Retention Times Reports (including acceptance limits and out-of-control flags)	VIII			X	X
	Reconstructed Ion Chromatogram for each sample and rerun, blank, spike, duplicate, and standard					X
	Raw Quantitation Report					X
	Raw and background subtracted mass spectra for each target analyte found					X
	Mass spectra of TICs with library spectra of 10 best-fit matches - Not required unless specified in project instructions					X
	Evidence of Manual Intergrations					X
	Copies of Sample Preparation Bench Sheets				X	X
Copies of Standard Preparation Logs					X	
Copies of Run Logs					X	
EDD				X	X	X

DATA DELIVERABLE PACKAGE REQUIREMENTS

Method	Deliverable Requirement	Equivalent EPA Form	Preliminary Results	CCI Level A	CCI Level B	CCI Level C
Metals	Case Narrative (See Note 1)			X	X	X
	Corrective Action Report			X	X	X
	Cross-reference of CCI Sample Numbers, Lab IDs, and analytical QC batches		X	X	X	X
	Chain-of-Custody Form, Cooler Receipt form		X	X	X	X
	Data Summary (Form 1) for Each Sample (See Note 2)	I-IN	X	X	X	X
	Blank Spike or Lab Control Sample (LCS) results (including concentration spiked, percent recovered, percent recovery acceptance limits) Note: The LCS shall be spiked with all analytes of interest.	VII-IN	X	X	X	X
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Report (including concentration spiked, percent recovered, percent recovery acceptance limits, relative percent difference (RPD), and RPD acceptance limits) Note: MS/MSD must be performed on each batch of 20 Navy samples. Matrix spike QC must contain all the targeted analytes of interest at a concentration not less than 10 times the MDL.	V (PART 1)IN	X	X	X	X
	Dilution Test	VIII-IN		X	X	X
	Post-digestion Spike Recovery for ICP only	V (PART 2)IN		X	X	X
	Recovery test for GFAA only			X	X	X
	Duplicate Sample Report			X	X	X
	Blank Results(Prep, ICB, CCB)	III-IN	X	X	X	X
	Initial Calibration Data	III-IN			X	X
	Second Source Verification Note: Result should be reported in terms of per cent recovery failing within (75-125 %)				X	X
	Continuing Calibration Data Note: All analytes must meet calibration acceptance criteria prior to analysis of samples.	II (PART I)-IN			X	X
	ICP Interference Check Sample Report	II (PART I)-IN			X	X
	Standard Addition Results	IV-IN			X	X
	Instrument and Method Detection Limit Summary				X	X
	Linear Range Summary					X
	Copies of Preparation Logs	IX-IN			X	X
Copies of Analysis Run Logs	XIII-IN			X	X	
Copies of Standard Preparation Logs	XIV-IN				X	
Raw Data and Instrument Printouts					X	
EDD				X	X	X

DATA DELIVERABLE PACKAGE REQUIREMENTS

Method	Deliverable Requirement	Equivalent EPA Form	Preliminary Results	CCI Level A	CCI Level B	CCI Level C
Inorganic Chemistry (Note 3)	Case Narrative (See Note 1)			X	X	X
	Corrective Action Report			X	X	X
	Cross-reference of CCI sample numbers, Lab IDs, and analytical QC batches		X	X	X	X
	Chain-of-Custody Form, Cooler Receipt form		X	X	X	X
	Data Summary (Form 1) for each blank and sample (See Note 2)		X	X	X	X
	Blank Spike or Lab Control Sample (LCS) results (including concentration spiked, percent recovered, percent recovery acceptance limits) Note: The LCS shall be spiked with all analytes of interest		X	X	X	X
	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Report (including concentration spiked, percent recovered, percent recovery acceptance limits) Note: MS/MSD must be performed on each batch of 20 Navy samples. Matrix spike QC must contain all the targeted analytes of interest at a concentration not less than 10 times the MDL.		X	X	X	X
	Duplicate Sample Report			X	X	X
	Calibration Reports Initial and Continuing Note: All analytes must meet calibration acceptance criteria prior to analysis of samples					X
	Second Source Verification (including acceptance limits) Note: Result should be reported in terms of per cent recovery failing within (75-125 %)				X	X
	Copies of Sample Preparation logs				X	X
	Raw Data and Instrument Printouts					X
	EDD				X	X

Notes:

- 1) Case narrative must include: Project summary referencing the analytical methodology, discussion of all protocol/procedure deviations, QC issues encountered during analyses and corrective actions taken as a result, summary and discussion of samples that were diluted because of the presence of an interference, non-target analyte, or target analyte, and discussion of QC samples exceeding established control limits and corrective actions taken.
- 2) Must include: Sample ID, Lab ID, date/time sampled, date received, extracted/analyzed Reporting Limit, Method Detection Limit, Dilution Factor, comments, results and qualifiers.
- 3) Deliverables depend on method required QC.
- 4) Laboratory should use in-house quality control acceptance criteria if the window is less than or equal to 60 point window (ex 75 125). Laboratory should indicate at the time of bid, which will be provided.
- 5) Laboratory should provide evidence at the time of bid of its ability to *routinely* meet contractual regulatory guidance criteria.(i.e. FL GCTL)

SAMPLE NO.

[Empty box for sample number]

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____

Sample wt/vol: _____ (g/ml) _____ Lab Sample ID: _____

Level: (low/med) _____ Lab File ID: _____

% Moisture: not dec. _____ Date Collected: _____ Time: _____

GC Column: _____ ID: _____ (mm) _____ Date Received: _____

Instrument ID: _____ Date Analyzed: _____ Time: _____

Soil Extract Volume: _____ (μ L) _____ Dilution Factor: _____ Analyst: _____

Soil Aliquot Volume: _____ (μ L) _____ Prep Batch: _____ Analytical Batch: _____

CONCENTRATION UNITS: _____

Analytical Method: _____

CAS NO.	COMPOUND	RESULT	Q	MDL	RL
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Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E524 E624 SW-846 8260B OLM04.1 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/SBRAC

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (14d/7d) <input type="checkbox"/> pH <2 <input type="checkbox"/> pH >2	<input type="checkbox"/> HT Met <input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A <input type="checkbox"/>
Surrogates (SS): 1, 2	Soil, low (2d/14d) <input type="checkbox"/> Preserved	<input type="checkbox"/> HT Met <input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A <input type="checkbox"/>
	<input type="checkbox"/> Correct surrogates <input type="checkbox"/> N/A <input type="checkbox"/> No*	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
	<input type="checkbox"/> Recov. OK <input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out <input type="checkbox"/> See Surr Wksht	<input type="checkbox"/>
MS/MSD or MS/LD: 3			
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> MS/MSD <input type="checkbox"/> MS/LD <input type="checkbox"/> None *	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
<input type="checkbox"/> Partial list	<input type="checkbox"/> %R OK <input type="checkbox"/> No *	<input type="checkbox"/> RPD OK <input type="checkbox"/> No *	<input type="checkbox"/> See Spike Wksht <input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> LCS/LCSD <input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met <input type="checkbox"/> No*	<input type="checkbox"/> See Spike Wksht <input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs			
PB, EB, FB/AB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht	<input type="checkbox"/>
MB, IB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht	<input type="checkbox"/>
Tune -BFB (TN) 5	<input type="checkbox"/> Frequency OK <input type="checkbox"/> Mass Assignment OK	<input type="checkbox"/> Ion Abundance OK <input type="checkbox"/> NO*	<input type="checkbox"/>
Calibration:			
Initial (IC) 6, 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht JUR
Continuing (CCV) 7, 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht <input type="checkbox"/>
Internal Standards (IS): 8	<input type="checkbox"/> IS used <input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/> IS out * <input type="checkbox"/>
Sample Evaluations (SAM) 1, 8, 10, raw	<input type="checkbox"/> All hits within cal. Range <input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows <input type="checkbox"/> No *	<input type="checkbox"/>
	<input type="checkbox"/> Mult Dilutions/Runs <input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.		<input type="checkbox"/>
	<input type="checkbox"/> Samples analyzed w/in 12hr clock		<input type="checkbox"/>
	<input type="checkbox"/> Samples bracketed by CCV <input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed	<input type="checkbox"/>
Field Duplicates (FD) 1	<input type="checkbox"/> Field Dup reported <input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met <input type="checkbox"/> No *	<input type="checkbox"/>

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E525 E625 SW-846 8270D OLM04.1 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/SBRAC

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out
	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
MS/MSD or MS/LD: 3	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK
<input type="checkbox"/> Partial list	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met
LCS (BS): 3	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
	<input type="checkbox"/> See Spike Wksht	<input type="checkbox"/> See Spike Wksht	<input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects
PB, EB, FB/AB	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects
MB, IB	<input type="checkbox"/> Frequency OK	<input type="checkbox"/> Mass Assignment OK	<input type="checkbox"/> Ion Abundance OK
Tune - DFTPP (TN)	<input type="checkbox"/> NO*	<input type="checkbox"/>	<input type="checkbox"/>
Calibration:	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
Initial (IC) 6, 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
Continuing (CCV) 7, 8	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met
Internal Standards (IS): 8	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows
Sample Evaluations (SAM)	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.	<input type="checkbox"/>
1, 8, 10, raw	<input type="checkbox"/> Samples analyzed w/in 12hr clock	<input type="checkbox"/>	<input type="checkbox"/>
	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed
Field Duplicates (FD)	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met
	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> No *

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E601 E602 SW-846 8021A SW-846 8015M Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/SBRAC

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

	Flags applied			
Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	_____	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	_____	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *	<input type="checkbox"/>
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (14d/7d) <input type="checkbox"/> pH <2 <input type="checkbox"/> pH >2	<input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A <input type="checkbox"/>
Surrogates (SS): 1, 2	Soil, low (2d/14d) <input type="checkbox"/> Preserved	<input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A <input type="checkbox"/>
MS/MSD or MS/LD: 3	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A <input type="checkbox"/> No*	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
Spiked: <input type="checkbox"/> All targets <input type="checkbox"/> Partial list	<input type="checkbox"/> MS/MSD <input type="checkbox"/> MS/LD <input type="checkbox"/> None *	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> %R OK <input type="checkbox"/> No *	<input type="checkbox"/> RPD OK <input type="checkbox"/> No *	<input type="checkbox"/> See Spike Wksht	<input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs PB, EB, FB/AB MB, IB	<input type="checkbox"/> LCS/LCSD <input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met <input type="checkbox"/> No*	<input type="checkbox"/> See Spike Wksht	<input type="checkbox"/>
Calibration: Initial (IC) 6, 8	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wrksht		<input type="checkbox"/>
Continuing (CCV) 7, 8	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wrksht		<input type="checkbox"/>
Internal Standards (IS): 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht	<input type="checkbox"/>
Sample Evaluations (SAM) 1, 8, 10, raw	<input type="checkbox"/> PID <input type="checkbox"/> EICD <input type="checkbox"/> FID	<input type="checkbox"/> Resolution criteria met	<input type="checkbox"/> No *	<input type="checkbox"/>
Field Duplicates (FD)	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht	<input type="checkbox"/>
	<input type="checkbox"/> IS used <input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/> IS out *	<input type="checkbox"/>
	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No * <input type="checkbox"/> Hits w/in RT windows	<input type="checkbox"/> No *	<input type="checkbox"/>
	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.		<input type="checkbox"/>
	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <40%D	<input type="checkbox"/> %D out <input type="checkbox"/> N/A	<input type="checkbox"/>
	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed	<input type="checkbox"/>
	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met <input type="checkbox"/> No *	<input type="checkbox"/>

QC Item	Comments	Flags
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Data Review and Validation for: GC VOC

Number of Samples: _____

Project Name & Task: _____

Project # & Case/SDG: _____

Organic Surrogate Worksheet

		Surrogate Compound & acceptance limits (%)					
Sample	Fraction	%	%	%	%	%	%

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E608 SW-846 8081B OLM03.2/OLM04.1 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/ _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

	Flags applied			
Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	_____	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	_____	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *	<input type="checkbox"/>
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*	<input type="checkbox"/>
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out	<input type="checkbox"/>
MS/MSD or MS/LD: 3	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *	<input type="checkbox"/>
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK	<input type="checkbox"/>
<input type="checkbox"/> Partial list	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met	<input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/>
PB, EB, FB/AB	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/>
MB, IB	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Resolution criteria met	<input type="checkbox"/>
Calibration:	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/>
Initial (IC) 6, 8	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/>
Continuing (CCV) 7, 8	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows	<input type="checkbox"/>
Internal Standards (IS): 8	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.		<input type="checkbox"/>
Sample Evaluations (SAM)	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <25/40%D	<input type="checkbox"/> %D out	<input type="checkbox"/>
1, 8, 10, raw	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed	<input type="checkbox"/>
Field Duplicates (FD)	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met	<input type="checkbox"/>

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E608 SW-846 8151A OLM03.2/OLM04.1 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/ _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

	Flags applied			
Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	_____	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	_____	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *	<input type="checkbox"/>
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*	<input type="checkbox"/>
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out	<input type="checkbox"/>
MS/MSD or MS/LD: 3	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *	<input type="checkbox"/>
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK	<input type="checkbox"/>
<input type="checkbox"/> Partial list	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met	<input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/>
PB, EB, FB/AB	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/>
MB, IB	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Resolution criteria met	<input type="checkbox"/>
Calibration:	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/>
Initial (IC) 6, 8	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/>
Continuing (CCV) 7, 8	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows	<input type="checkbox"/>
Internal Standards (IS): 8	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.		<input type="checkbox"/>
Sample Evaluations (SAM)	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <25/40%D	<input type="checkbox"/> %D out	<input type="checkbox"/>
1, 8, 10, raw	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed	<input type="checkbox"/>
Field Duplicates (FD)	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met	<input type="checkbox"/>

QC Item	Comments	Flags

Data Review and Validation for: **Polychlorinated Biphenyls (PCBs)**

Number of Samples: _____

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E608 SW-846 8082 OLM03.2/OLM04.1 Other _____

Program: AFCEE CLP NFESC Navy R/ _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met <input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
	Soil, low (14d/40d) <input type="checkbox"/> HT Met <input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A	<input type="checkbox"/>
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates <input type="checkbox"/> N/A <input type="checkbox"/> No*	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
	<input type="checkbox"/> Recov. OK <input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out <input type="checkbox"/> See Surr Wksht	<input type="checkbox"/>
MS/MSD or MS/LD: 3			
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> MS/MSD <input type="checkbox"/> MS/LD <input type="checkbox"/> None *	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
<input type="checkbox"/> Partial list	<input type="checkbox"/> %R OK <input type="checkbox"/> No *	<input type="checkbox"/> RPD OK <input type="checkbox"/> No *	<input type="checkbox"/> See Spike Wksht
LCS (BS): 3	<input type="checkbox"/> LCS/LCSD <input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met <input type="checkbox"/> No*	<input type="checkbox"/> See Spike Wksht
Blanks (BL): 1, 4, Ext Logs			
PB, EB, FB/AB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht	<input type="checkbox"/>
MB, IB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht	<input type="checkbox"/>
Calibration:			
Initial (IC) 6, 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht
	<input type="checkbox"/> # of peaks used acceptable	<input type="checkbox"/> No * <input type="checkbox"/> see comments regarding aroclor calib.	<input type="checkbox"/>
Continuing (CCV) 7, 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht
Internal Standards (IS): 8	<input type="checkbox"/> IS used <input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/> IS out *
Sample Evaluations (SAM)	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No * <input type="checkbox"/> Hits w/in RT windows	<input type="checkbox"/> No *
1, 8, 10, raw	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.	<input type="checkbox"/>
	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <25/40%D <input type="checkbox"/> %D out <input type="checkbox"/> N/A	<input type="checkbox"/>
	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No * <input type="checkbox"/> Manual integration performed	<input type="checkbox"/>
Field Duplicates (FD)	<input type="checkbox"/> Field Dup reported <input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met <input type="checkbox"/> No *	<input type="checkbox"/>

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E614 E622 SW-846 8141A Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/SBRAC _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A <input type="checkbox"/> No*	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out <input type="checkbox"/> See Surr Wksht
MS/MSD or MS/LD: 3	Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> MS/MSD <input type="checkbox"/> MS/LD <input type="checkbox"/> None *	<input type="checkbox"/> Lab Limits <input type="checkbox"/> Meth/Prog. Limits
	<input type="checkbox"/> Partial list	<input type="checkbox"/> %R OK <input type="checkbox"/> No *	<input type="checkbox"/> RPD OK <input type="checkbox"/> No *
LCS (BS): 3	<input type="checkbox"/> LCS/LCSD <input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met	<input type="checkbox"/> No* <input type="checkbox"/> See Spike Wksht
Blanks (BL): 1, 4, Ext Logs	PB, EB, FB/AB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht
	MB, IB	<input type="checkbox"/> Freq. OK <input type="checkbox"/> All ND <input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wksht
Calibration:	Initial (IC) 6, 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided <input type="checkbox"/> See Calib Wksht
	Continuing (CCV) 7, 8	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Resolution criteria met <input type="checkbox"/> No *
Internal Standards (IS): 8	Sample Evaluations (SAM)	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No * <input type="checkbox"/> Not provided <input type="checkbox"/> See Calib Wksht
	1, 8, 10, raw	<input type="checkbox"/> IS used <input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met <input type="checkbox"/> IS out *
		<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No * <input type="checkbox"/> Hits w/in RT windows <input type="checkbox"/> No *
		<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.
		<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <40%D <input type="checkbox"/> %D out <input type="checkbox"/> N/A
		<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No * <input type="checkbox"/> Manual integration performed
Field Duplicates (FD)		<input type="checkbox"/> Field Dup reported <input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met <input type="checkbox"/> No *

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: E610 SW-846 8100 SW-846 8310 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/ _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out
	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
MS/MSD or MS/LD: 3	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *
	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK
	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS	<input type="checkbox"/> All criteria met
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
	<input type="checkbox"/> See Spike Wksht		<input type="checkbox"/>
<input type="checkbox"/> Partial list	<input type="checkbox"/> See Spike Wksht		<input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> See Spike Wksht		<input type="checkbox"/>
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects
PB, EB, FB/AB	<input type="checkbox"/> See Blank Wksht		<input type="checkbox"/>
MB, IB	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects
Calibration:	<input type="checkbox"/> See Blank Wksht		<input type="checkbox"/>
Initial (IC) 6, 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Resolution criteria met
Continuing (CCV) 7, 8	<input type="checkbox"/> See Calib Wksht		<input type="checkbox"/>
Internal Standards (IS): 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met
Sample Evaluations (SAM)	<input type="checkbox"/> IS out *		<input type="checkbox"/>
1, 8, 10, raw	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows
	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.	<input type="checkbox"/>
	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> P/C ** <40%D	<input type="checkbox"/> %D out
	<input type="checkbox"/> Manual integration performed		<input type="checkbox"/>
Field Duplicates (FD)	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/>
	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met
			<input type="checkbox"/> No *

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: GRO SW-846 8100 SW-846 8310 Other _____

Program: AFCEE CLP NFESC Navy RAC III/IV/ _____

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

	Flags applied			
Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *		<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *		<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *	<input type="checkbox"/>
Holding Times (HT): 1, 4, COC, Ext. Logs	Water <input type="checkbox"/> Purge/Ext. HT Met	<input type="checkbox"/> Analysis HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
Surrogates (SS): 1, 2	Soil <input type="checkbox"/> Purge/Ext. HT Met	<input type="checkbox"/> Analysis HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
MS/MSD or MS/LD: 3	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*	<input type="checkbox"/> Lab Limits
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out	<input type="checkbox"/> See Surr Wksht
<input type="checkbox"/> Partial list	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *	<input type="checkbox"/> Lab Limits
LCS (BS): 3	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK	<input type="checkbox"/> No *
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS only	<input type="checkbox"/> All criteria met	<input type="checkbox"/> No*
PB, EB, FB/AB	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wrksht
MB, IB	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects	<input type="checkbox"/> See Blank Wrksht
Calibration:	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht
Initial (IC) 6, 8	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Resolution criteria met	<input type="checkbox"/> No *
Continuing (CCV) 7, 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided	<input type="checkbox"/> See Calib Wksht
Internal Standards (IS): 8	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met	<input type="checkbox"/> IS out *
Sample Evaluations (SAM)	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows	<input type="checkbox"/> No *
1, 8, 10, raw	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.		
Field Duplicates (FD)	<input type="checkbox"/> Fingerprinting requested	<input type="checkbox"/> Fingerprinting evaluated	<input type="checkbox"/> ID correct	<input type="checkbox"/> N/A
	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed	
	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met	<input type="checkbox"/> No *

QC Item	Comments	Flags

Project Name & Task: _____

Project # & Case/SDG: _____

Methods: SW-846 8280 SW-846 8290 Other: _____

Program: AFCEE CLP NFESC Navy RAC III/IV/SBF

Field QC Samples: _____

Reviewed by & Date: _____

Matrix: Water Soil/Solid TCLP/SPLP Other: _____

Case Narrative: No exceptions not provided Exceptions: _____

Flags applied

Data Pkg (DP): pkg., COC, invoice	<input type="checkbox"/> All required deliverables in pkg.	<input type="checkbox"/> Deliverables Missing *	<input type="checkbox"/>
	<input type="checkbox"/> All samples on COC reported	<input type="checkbox"/> Samples not reported *	<input type="checkbox"/>
	<input type="checkbox"/> Invoice received/correct	<input type="checkbox"/> Receipt temperatures ok	<input type="checkbox"/> No *
Holding Times (HT): 1, 4, COC, Ext. Logs	Water (7d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
	Soil, low (14d/40d) <input type="checkbox"/> HT Met	<input type="checkbox"/> HT Exceeded *	<input type="checkbox"/> N/A
Surrogates (SS): 1, 2	<input type="checkbox"/> Correct surrogates	<input type="checkbox"/> N/A	<input type="checkbox"/> No*
	<input type="checkbox"/> Recov. OK	<input type="checkbox"/> 1 or more recov.out *	<input type="checkbox"/> Surr. Diluted out
MS/MSD or MS/LD: 3	<input type="checkbox"/> MS/MSD	<input type="checkbox"/> MS/LD	<input type="checkbox"/> None *
Spiked: <input type="checkbox"/> All targets	<input type="checkbox"/> %R OK	<input type="checkbox"/> No *	<input type="checkbox"/> RPD OK
<input type="checkbox"/> Partial list	<input type="checkbox"/> Lab Limits	<input type="checkbox"/> Meth/Prog. Limits	<input type="checkbox"/>
LCS (BS): 3	<input type="checkbox"/> LCS/LCSD	<input type="checkbox"/> LCS	<input type="checkbox"/> All criteria met
Blanks (BL): 1, 4, Ext Logs	<input type="checkbox"/> See Spike Wksht	<input type="checkbox"/>	<input type="checkbox"/>
PB, EB, FB/AB	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> All ND	<input type="checkbox"/> Blanks have detects
MB, IB	<input type="checkbox"/> See Blank Wrksht	<input type="checkbox"/>	<input type="checkbox"/>
Tune (TN) 5	<input type="checkbox"/> Freq. OK	<input type="checkbox"/> Mass Assignment OK	<input type="checkbox"/> Ion Abundance OK
Calibration:	<input type="checkbox"/> NO*	<input type="checkbox"/>	<input type="checkbox"/>
Initial (IC) 6, 8	<input type="checkbox"/> # of Levels/Linearity Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
	<input type="checkbox"/> Degradation Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Resolution criteria met
Continuing (CCV) 7, 8	<input type="checkbox"/> Correct Frequency/Criteria Met	<input type="checkbox"/> No *	<input type="checkbox"/> Not provided
Internal Standards (IS): 8	<input type="checkbox"/> See Calib Wksht	<input type="checkbox"/>	<input type="checkbox"/>
Sample Evaluations (SAM) 1, 8, 10, raw	<input type="checkbox"/> IS used	<input type="checkbox"/> No IS	<input type="checkbox"/> All samples/stds. IS criteria met
	<input type="checkbox"/> All hits within cal. Range	<input type="checkbox"/> No *	<input type="checkbox"/> Hits w/in RT windows
	<input type="checkbox"/> Mult Dilutions/Runs	<input type="checkbox"/> Data rejected - only 1 valid result for each parameter/samp.	<input type="checkbox"/>
	<input type="checkbox"/> Confirmation analyzed/reported	<input type="checkbox"/> N/A	<input type="checkbox"/>
	<input type="checkbox"/> Samples bracketed by CCV	<input type="checkbox"/> No *	<input type="checkbox"/> Manual integration performed
Field Duplicates (FD)	<input type="checkbox"/> Field Dup reported	<input type="checkbox"/> N/A	<input type="checkbox"/> Precision criteria met
	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> No *

QC Item	Comments	Flags

Data Review and Validation for Dioxins Number of Samples: _____

Project Name & Task: _____

Project # & Case/SDG: _____

Data Review and Validation for:

Air Sample Analysis

Number of Samples:

Project Name & Task:

Project # & Case/SDG:

Methods:

EPA 18

TO-

GC

GC/MS

Other:

Program:

AFCEE

CLP

NFESC

Navy RAC III/IV/

Field QC Samples:

Reviewed by & Date:

Matrix:

Tedlar Bag

Suma Canister

Tube Other:

Case Narrative:

No exceptions

not provided

Exceptions:

Flags applied

Data Pkg (DP):

pkg., COC, invoice

All required deliverables in pkg.

Deliverables Missing *

All samples on COC reported

Samples not reported *

Invoice received/correct

Receipt temperatures ok

No *

Holding Times (HT):

1, 4, COC, Ext. Logs

Surrogates (SS): 1, 2

Tedlar (72hrs)

HT Met

HT Exceeded *

N/A

Suma (7/14d)

HT Met

HT Exceeded *

N/A

Correct surrogates

N/A

No*

Lab Limits

Meth/Prog. Limits

Recov. OK

1 or more recov.out *

Surr. Diluted out

See Surr Wksht

MS/MSD or MS/LD: 3

Spiked:

All targets

Partial list

MS/MSD

MS/LD

None *

Lab Limits

Meth/Prog. Limits

%R OK

No *

RPD OK

No *

See Spike Wksht

LCS (BS): 3

LCS/LCSD

LCS

All criteria met

No*

See Spike Wksht

Blanks (BL): 1, 4, Ext Logs

PB, EB, FB/AB

Freq. OK

All ND

Blanks have detects

See Blank Wrksht

MB, IB

Freq. OK

All ND

Blanks have detects

See Blank Wrksht

Calibration:

Initial (IC) 6, 8

of Levels/Linearity Criteria Met

No *

Not provided

See Calib Wksht

PID

EICD

FID

MS

Resolution criteria met

No *

Continuing (CCV) 7, 8

Correct Frequency/Criteria Met

No *

Not provided

See Calib Wksht

Internal Standards (IS): 8

IS used

No IS

All samples/stds. IS criteria met

IS out *

Sample Evaluations (SAM)

1, 8, 10, raw

All hits within cal. Range

No *

Hits w/in RT windows

No *

Mult Dilutions/Runs Data rejected - only 1 valid result for each parameter/samp.

Confirmation analyzed/reported

P/C ** <40%D

%D out

N/A

Samples bracketed by CCV

No *

Manual integration performed

Field Duplicates (FD)

Field Dup reported

N/A

Precision criteria met

No *

QC Item

Comments

Flags

Laboratory Quality Assurance Program Plan and Custody Seal



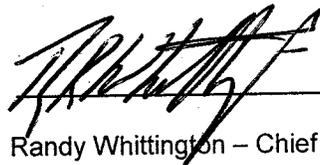
QUALITY ASSURANCE PROGRAM PLAN

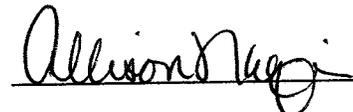
FOR

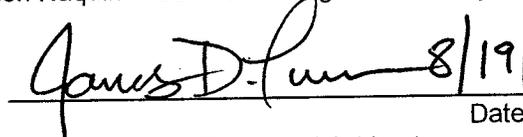
**GCAL Inc.
7979 GSRI Avenue
Baton Rouge, Louisiana 70820
225-769-4900**

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August 20, 2010**

Approval Signatures:

 08/19/10
Date
Randy Whittington – Chief Executive Officer

 8/19/10
Date
Allison Naquin – General Manager/Laboratory Manager

 8/19/10
Date
James Turner – QA Director

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1 Quality Assurance Policy Statement

Quality Assurance consists of a planned system of activities necessary to provide confidence in the results of laboratory analytical determinations. The principal objective of GCAL is the production of high quality analytical data through the use of measurements that are accurate and reliable for the intended purpose. We are dedicated to providing analytical data and services that conform to all of the requirements specified and expected by our clients. This Quality Assurance Program Plan (QAPP) details facilities, personnel and equipment necessary for accomplishing this objective along with general procedures and practices that will be followed to maintain adherence to the objective. All policies and procedures have been structured in accordance with the NELAC standards, the DOD Quality Systems Manual, and in accordance with applicable state, EPA, and other regulatory agency requirements, regulations, methods, and guidance. GCAL's management staff is dedicated to maintaining compliance with both the NELAC Standard and the DOD Quality Systems Manual.

There is a commitment and dedication by all laboratory staff to produce data of known and documented quality. This commitment and dedication to quality is fully supported from the bench level to upper management in order to meet the objectives of our laboratory and best serve our clients.

GCAL's approach to Quality Assurance starts with the General Manager who delineates policy and sets goals in conjunction with senior management personnel. Management staff and laboratory personnel implement policies. All departments are involved in the process by providing assessment of operating procedures along with recommendations for improvements or corrections. The QAPP and the appropriate Standard Operating Procedures are distributed to all laboratory personnel as controlled documents according to SOP QA-001 (Document Control). All personnel are required to read and comply with this program.

The Quality Assurance/Quality Control Director oversees prevention, assessment, and correction procedures for the analytical laboratory and various associated departments within the organization. These three functions; prevention, assessment, and correction, comprise the foundation of the laboratory's approach to Quality Assurance. Through this foundation, GCAL's management staff is committed to continually improve the quality system.

Prevention covers positive actions taken before or during analyses to insure that the analytical systems are functioning properly. Prevention includes such things as instrument calibration and maintenance, frequent standardization, personnel training and quality control planning.

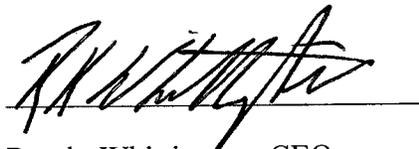
Assessment is a component of quality control that includes monitoring of performance to determine precision and accuracy. Examples include duplicate and spike analyses, check samples, peer review of calculations and validation of methodology.

Correction is action taken to determine the causes of quality defects and to restore proper functioning of the analytical system. This includes trouble shooting to correct instrument malfunctions, or retraining of personnel.

All quality assurance activity requires constant monitoring and documentation to provide evidence of consistent, valid analytical data. GCAL keeps records of such activities in order to have available for its clients documented assurance that the data they receive quantitatively reflect the parameters requested.

The policies and practices of quality assurance/quality control presented in this plan are set forth as minimums. Additional quality assurance/quality control measures are defined by a specific project plan.

In the case of discrepancies between this document and SOPs, the SOP shall take precedence. A list of supporting SOPs including technical procedures are located in Appendix F of this document.

A handwritten signature in black ink, appearing to read 'Randy Whittington', is written over a horizontal line.

Randy Whittington, CEO

2 Ethics Policy

GCAL utilizes a clearly stated ethics policy in the form of the following Ethics and Data Integrity Statement. This agreement is discussed with all new employees during orientation and is then signed and retained with the employee's training file. Violation of the agreement is basis for termination of employment. Employees receive training in data integrity annually. Each employee signs the ethical policy yearly.

GCAL

ETHICS AND DATA INTEGRITY AGREEMENT

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at GCAL. Our core values are honesty, success, service and integrity. I understand that it is critical for our long-term success that each and every employee aligns with all company core values.

I agree that in the performance of my duties for GCAL and its clients, I shall conform to the following ethics standards and will report immediately to the Quality Assurance Manager and the appropriate supervisor any information concerning misrepresentation of analytical data that includes, but is not limited to:

- 1) Altering an instrument computer or clock for any inappropriate purpose;
- 2) Altering the contents of logbooks and/or data sheets to misrepresent data;
- 3) Misrepresenting an analyst's identity;
- 4) Changing raw data documents with correction fluid;
- 5) Preparation and submittal of "fake" data packages;
- 6) Inappropriate calibration techniques such as peak shaving, setting fraudulent integrator parameters, or use of computer macros that alter QC results.
- 7) Changing reported results without proper documentation and approval;
- 8) Altering injection volumes for calibration and misrepresenting the true values;
- 9) Failure to comply with standard operating procedures or methods without proper documentation and approval;
- 10) Any attempt to misrepresent data or events as they actually occur in the course of data production, review or reporting;

- 11) Disposing of or deleting electronic data files or hardcopy of raw data;
- 12) All QC and PT samples must be analyzed in the same manner as client samples. This includes glassware selection, all prep steps, any clean up steps, reagent addition, and analysis, unless specified to be different by the SOP or reference method;
- 13) Engaging in any practice that ultimately misrepresents data or narratives in any way.

I will not knowingly participate in any such activity and will not tolerate unethical practices by others. I understand that confidentiality will be strictly enforced by GCAL when dealing with these matters. As a further extension of my commitment to this program, I am responsible for seeking approval to report data resulting from techniques or procedures that deviate from standard operating procedures, methods, or industry standard practices. Any such reporting of data will include a laboratory narrative that must be approved by the appropriate supervisor and the QA Director.

If I am unsure of how to properly handle data generated by me, I am responsible for seeking advice and approval from the Quality Assurance Director and the appropriate supervisor. I agree to inform the Quality Assurance Director and the appropriate supervisor of any accidental reporting of non-authentic data by others or myself within 24 hours of discovery.

I understand that if I knowingly participate in any such prohibited activity, I will be subject to serious disciplinary action that may include termination by GCAL. I also understand that I face individual suspension and debarment from all Federal programs should I be convicted of such practices. I understand that suspension and debarment from all Federal programs affects my ability to work in the environmental field, as well as, any other professions where government funding or loans may be involved. I understand the most serious consequence of unethical conduct can be imprisonment if convicted.

However, it is not the company's intent to punish anyone for an accidental mistake or oversight. Employees will not face disciplinary actions in this case. Repeated careless or neglectful behavior will be subject to corrective action. Covering up a mistake or oversight is not acceptable behavior and will result in termination. Mistakes or oversights are immediately reported to the Quality Assurance Director and the appropriate supervisor.

My signature affirms my understanding of the consequences of violating this "ETHICS AND DATA INTEGRITY AGREEMENT" and my commitment to its intent. My signature further affirms that I have received formal training on this topic.

(Signature)

(Date)

3 Administrative Organization

GCAL is organized along clear lines of authority to provide our clients with service that is efficient and reliable. The organizational structure of the laboratory is shown in Appendix A.

It is the policy of the laboratory that at each management and operational level a designated deputy or deputies will maintain continuity of service and other functions in the event of absence of key staff. The deputies are responsible for the completion of duties during the staff member's absence. Louisiana DEQ, LELAP must be notified in the instance of the Laboratory Manager's absence of more than 65 days.

Position	Deputy
General Manager	Technical Services Manager
Laboratory Manager (Technical Director)	General Manager
QA/QC Director	General Manager
Department Supervisor	Group Leader
IT Manager	Laboratory Manager and CEO

To ensure communication within the laboratory and to communicate project requirements to the analyst, the laboratory supervisors, report generation, and the Laboratory Manager meet daily with project management to discuss key issues for that day.

3.1 Roles, Responsibilities, and Qualifications

The following lists the general roles and responsibilities in each level of the laboratory. Resumes of key personnel are attached in Appendix B.

- 3.1.1 The CEO directs the functional areas of marketing and finance.
- 3.1.2 The General Manager bears the primary responsibility for data quality at the laboratory. The General Manager directs laboratory policies.
- 3.1.3 The Laboratory Manager (Technical Director) is responsible for coordinating the activities of all laboratory personnel. The Laboratory Manager assures the commitment of sufficient resources for the timely generation of data of a known quality. Duties include monitoring standards of performance in quality control and quality assurance and monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data. The Laboratory Manager must have at least a bachelor's degree in Chemical, Environmental, Biological or Physical sciences, or Engineering, with at least 24 hours of chemistry, and at least 2 year experience in environmental laboratory testing. A master's or doctoral degree may be substituted for

one year of experience. In the event that the Laboratory Manager is absent for a period of time exceeding 15 consecutive calendar days, they will designate another full-time staff member meeting the above qualifications to temporarily perform their duties. If this absence is to exceed 65 consecutive calendar days, all accreditation bodies will be notified in writing.

- 3.1.4 The Technical Services Manager is responsible for coordinating the activities of the sample administration department, client services, and administrative support personnel.
- 3.1.5 The Information Technology Director manages the implementation and development of information technology tools. IT Director is also responsible for the automated data collection systems used by the laboratory. IT Director performs strategic planning for IT projects based on projected needs of the Laboratory. Interacts with clients to determine IT requirements such as electronic deliverables.
- 3.1.6 The Data Validation Manager is responsible for report validation and review. The Data Validation Manager is also responsible for review of Quality Assurance Project Plans on incoming projects and implementation of such plans throughout the laboratory. Assists the lab in method implementation and development. Additional duties include advising the laboratory on reference methods and improving method performance. The Technical Director is also responsible for review of final reports. Any discrepancies found in the data are reported to the appropriate Department Supervisor for review and correction if necessary.
- 3.1.7 The QA/QC Director is responsible for the preparation and maintenance of the laboratory Quality Assurance Program Plan. The QA/QC Director acts as the official laboratory contact for audits, performance evaluation studies, and project-specific quality control issues. The QA/QC Director approves and confirms the implementation of corrective actions. The QA/QC Director is responsible for the approval and distribution of controlled documents. The QA/QC Director has the authority to intercede in all areas where quality related problems exist. No work will be released until the related quality deficiency has been corrected and approval has been given to proceed forward. The QA/QC Director reports directly to the General Manager, and is not in the chain of command of the departments audited. It is the policy of the GCAL that the QA/QC Director operates independent of the production pressures of the laboratory. The QA/QC Director must have training in QA/QC and a general knowledge of the test under the scope of accreditation. In addition, the QA/QC Director is responsible for implementing, maintaining, and improving the quality system; ensuring that all personnel understand their contributions to the quality system; ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system; evaluating the effectiveness of training; and using available tools, such as audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and

management reviews in efforts to monitor trends and continually improve the quality system.

- 3.1.8 Department Supervisors are responsible for the overall flow of work and data through the laboratory. They are responsible for the maintenance of accurate SOP's with the input of the QA/QC Department. Further responsibilities include management of all activities within their department, ensuring that all instrumentation and equipment meet performance criteria and calibration requirements, and training of laboratory staff. The Supervisor is responsible for validating data released from the department. Department Supervisors inform the Laboratory Manager or Technical Services Manager of project status and capacity issues. Department supervisors must have experience in the methods performed in their department and with data validation.
- 3.1.9 Project Managers act as liaisons between the laboratory and the client. Responsibilities include sample scheduling, communicating project-specific requirements to laboratory personnel, review of log-in summaries, notifying the client of any sample receipt or analytical problems, monitoring the progress of analytical work, and providing data to clients in a timely manner. Project Managers document client complaints.
- 3.1.10 Analysts/chemists are responsible for the generation of data by analyzing samples according to written SOP's. They are also responsible for ensuring that all documentation related to the analysis is accurate and complete. The analyst/chemist shall inform the Department Supervisor of quality problems immediately. The analysts/chemists have the authority to accept or reject data based on compliance with QC acceptance criteria. Analysts/Chemists are responsible for initial review of all data.
- 3.1.11 Group Leaders guide the scheduling of sample analysis, ensure there is sufficient staff available, and perform other duties as directed by the Department Supervisor. The group leader shall act as the Department Supervisor in the event of absence.
- 3.1.12 Safety Compliance Officer is responsible for training lab personnel in proper safety procedures. Safety is responsible for checking and maintaining safety equipment, maintaining documents regarding waste disposal, safety audits, and MSDS files.

3.2 Personnel Training

It is the policy of GCAL to hire employees with an educational background and/or experience in an analytical field. On-the-job training takes place for all new employees based on needs identified by the job description and tasks of the position.

- 3.2.1 Training Program - The training program begins with an orientation program designed to familiarize the new employee with safety and chemical hygiene issues, the importance of QA/QC in the analytical laboratory, general laboratory procedures, and company policies. All employees undergo training in ethical and legal responsibilities including the

potential penalties for improper, unethical, or illegal actions. Each employee must read and sign an Ethics and Data Integrity Agreement. All technical personnel undergo a training process involving twelve lecture tapes covering basic laboratory functions. A written test follows each lecture tape. Employees who perform or review manual integrations as part of their job functions are also trained in manual integration policy and review and sign a manual integration policy statement.

- 3.2.2 New employees are under the supervision of experienced analysts and/or the department supervisors who are responsible for showing them the analytical procedures including the applicable QA/QC. This training includes review of the Standard Operating Procedure (SOP), reference methods, and hands-on training with instrumentation or equipment. Manuals for various methods are available to laboratory personnel. Among the manuals are current copies of the EPA Test Methods for Evaluating Solid Waste (SW846), Chemical Analysis for Water and Wastes, Standard Methods, and ASTM methods. The analyst will perform an acceptable initial demonstration of capability before being allowed to analyze and post results without direct supervision. This is accomplished by analyzing four laboratory control samples (LCS) and verifying achievement of acceptable precision and accuracy requirements of the laboratory. This demonstration will be repeated whenever there is a significant change to the instrument or test method and annually. New employees are hired for a probationary period of three months. At the end of three months the employee's records are reviewed and evaluated for performance and productivity and a decision is made whether to continue employment. Additional information on DOC is described in SOP QA-014.
- 3.2.3 Ongoing training - GCAL also recognizes development training as a means to increase the effectiveness of the employee and the organization. Therefore, GCAL utilizes other training methods along with on-the-job training. Examples of this are seminars, specialized training by instrument manufacturers, internal training courses, and encouraging the employees to take related college courses. On-going proficiency is documented by performance evaluation samples, an annual demonstration of capability, and /or analysis of blind samples.
- 3.2.4 Additional training - Training is also necessary for an employee whose performance does not meet standard requirements. This deficiency is identified in a performance appraisal or through the occurrence of problems.
- 3.2.5 Periodic reviews are given to all personnel. The purpose of these reviews is to give recognition for good work and outline personnel and departmental objectives, suggestions for improvement and clarification of responsibilities. Other topics that concern either employee or employer are also discussed at this time.

3.3 Training Files

The QA/QC Department maintains training files for each employee. The records include the demonstration of capability, training course certificates, in-house or external training seminar documentation, and ethics and manual integration agreements.

4 Quality Assurance

4.1 Quality Assurance Responsibilities

The direct and ultimate responsibility for assuring data quality at GCAL rests with the General Manager. The General Manager develops policies and general quality assurance strategies in collaboration with the Management Staff and Department Supervisors.

GCAL has clearly defined staff Quality Assurance (QA) responsibilities. The first level of QA lies with the laboratory analyst, who is responsible for performing the work properly, documenting it, and obtaining peer review to assure that it meets scientific standards. To accomplish this, the analyst must have a clear understanding of the analytical techniques and procedures used and the factors that affect the quality of the results. Analysts' capabilities are verified prior to conducting analyses and reviewed periodically thereafter.

Analysts must have a working knowledge of the QA policies, including data quality objectives for laboratory control standards, duplicates, and spikes; an understanding of detection limits and standard calibration requirements; and knowledge of preventive maintenance techniques. It is a requirement that all staff review and follow all applicable SOPs and this Quality Assurance Program Plan.

The second level of quality assurance lies with the management staff and Department Supervisors. Management is responsible for the proper training of analysts and stresses the importance of accuracy and reliability of results. Management is responsible for the quality of all analytical data produced. This responsibility includes routine review and approval or disapproval of all data and inspection of the QC records associated with the data. If the data are not adequately substantiated, corrective action is taken.

The QA/QC Director supports the entire process of the QA/QC program. This includes administration of the program as outlined in this manual, maintenance of QA records including this QAPP, and preparation of reports to management covering QA activities. The QA/QC Director or designee performs periodic audits of the QA procedures and staff for all departments, establishes and maintains accreditation for regulators, and coordinates all performance audits (e.g. check sample programs). The QA/QC Director also oversees the corrective action program.

4.2 Performance audits (PT studies)

GCAL participates in at least two Water Pollution studies, two UST studies, and two Hazardous Waste studies annually. The QA/QC Department orders the necessary studies at approximately six-month intervals from a NVLAP approved vendor. When samples are received they are logged into the LIMS as a work order. All PT studies are analyzed as client samples with the normal batch QC performed. It is the policy of GCAL that PT studies are handled as client samples throughout the analytical process. It is inappropriate to analyze multiple duplicates or dilutions unless this procedure is described in the approved analytical SOP as the required

analytical method for all samples. Any problems with PT samples shall be immediately communicated to the QA/QC Department.

Samples are reviewed in a different manner than client samples. The QA/QC Department is responsible for the final review and reporting of PT results to the PT provider. PT results and batch QC is reviewed carefully. Also reviewed are sample prep for appropriate dilution, reported concentration, consistency across methods, logical results, and transcription errors. Batch QC failures will necessitate re-analysis of the PT sample, or comments in the case narrative. If, in the QA/QC Director's opinion, GCAL must withdraw from a PT study, the primary accrediting authority shall be informed in writing. Results are reported to the PT provider, generally by fax or electronic submission.

The QA/QC Director shall review scored results for performance and accuracy. Results scored "Not Acceptable" shall be thoroughly reviewed to determine the cause of the failure and corrective action performed. In all cases a corrective action (rapid response) PT sample or blind QC sample shall be analyzed for any failed method/analyte/matrix. Documentation of scored PT results, raw data, and corrective action shall be kept on file. In addition, once root cause analysis, corrective action and additional proficiency testing are complete, this information is submitted to LDEQ, ACLASS, and all other pertinent accrediting bodies that require this information.

It is inappropriate for any personnel to share results or to attempt to obtain results from any other participating laboratory or PT provider.

GCAL utilizes the first WP Study of the year to meet DMR requirements for clients that use GCAL for analysis of samples regulated by a discharge permit. Samples are analyzed, reviewed, and the applicable results are reported to the client. It is the intention of GCAL to submit results to the client at least one-calendar week before results are due to the DMR provider.

It is the responsibility of the QA/QC Director to maintain compliance with NELAC and DOD regulations regarding PT analysis and reporting. Compliance includes passing two of every three studies and performing PT studies for every matrix/method/analyte offered by approved PT providers. Experimental studies shall be performed as required. The QA/QC Department shall suspend any matrix/method/analyte combination that fails two of the three most recent PT study until the laboratory meets the requirements of initial acceptability. See SOP QA-015 for more details.

4.3 Accreditation

GCAL is an accredited NELAC laboratory (Certificate number 01955). The primary accrediting authority is the Louisiana Environmental Laboratory Accreditation Program (LELAP) administered through Louisiana Department of Environmental Quality. A full list of accreditations held is maintained in the QA Department and is listed in Appendix E.

Compliance with and maintenance of laboratory accreditation is the responsibility of the QA/QC Director. Scope of application and certificates shall be kept on file and scanned and stored electronically. All correspondence with accrediting authorities shall be kept on file.

Maintenance of accreditation is based on compliance with NELAC Chapter 5 and associated appendices. It is the responsibility of the QA/QC Director to read and implement changes when each addition is approved for use.

4.4 Quality Control

This section describes the types of quality control samples used in the laboratory and how they are used to assess data precision and accuracy. When the analysis of a sample set is completed, the results will be reviewed and evaluated to assess the validity of the data set. All QC is processed and analyzed using the same conditions as the samples.

4.4.1 A reagent and/or method blank is prepared and analyzed with each set of samples. Field blanks (if provided by the client) are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air on site, during shipment, and in the laboratory. Storage blanks are also used in volatile refrigerators and analyzed every other week. The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents shall be checked for contamination and the analysis halted until the system can be brought under control before further sample analysis proceeds. The concentration of an analyte in a reagent blank must be less than $\frac{1}{2}$ the reporting limit or less than 5% of the analyte detected in the associated samples. Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis will be reviewed. If the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOA's, ambient air in the laboratory and reagents shall be checked as possible sources of contamination. If method blanks are not acceptable, the associated samples must be re-prepped and analyzed. For storage blank criteria, please see SOP GEN-010 for details.

4.4.2 A Laboratory Control Standard (LCS) consisting of an interference free matrix spiked with the analytes of interest, or a representative list of the analytes of interest, is prepared and analyzed with each batch of twenty or fewer samples. Some projects, such as DOD, require that all target analytes be spiked. This information is documented by the project manager as a comment in the profile. Analysts are responsible for checking comments in their workstation. Analyte-free reagent water is used for water samples. A purified solid matrix such as Ottawa sand, Teflon beads, or sodium sulfate is used for soil or solid samples. For those tests that it is difficult to obtain a suitable solid matrix for spiking, analyte free reagent water is taken through the preparation and analysis procedure. A

standard reference material is allowable for use as an LCS. The results of check standard analyses are compared with the true values and the percent recovery of the check standard is calculated. If correction is required, the check standard is reanalyzed to demonstrate that the corrective action has been successful. Acceptable accuracy (control limits) is a requirement of the method or determined by the laboratory by the use of control charts.

- 4.4.3 A matrix spike (a sample to which known concentrations of target analytes have been added before sample manipulation) is performed on one sample in each batch of twenty or fewer samples for those tests that spiking is applicable. The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. If the accuracy value is outside the control limits for the given parameter, the LCS is reviewed to verify the analytical system is in control. The failure is a result of an error or the matrix. If the accuracy value is outside the control limit, the sample set (parent sample, MS/MSD, or duplicate) is reanalyzed for the parameter in question, unless insufficient sample volume is available or the samples are past holding time. Generally, matrix spike control limits are set as the LCS control limits. Use the same spike list and concentration as required in the LCS.
- 4.4.4 A duplicate for each matrix type is included in each batch of twenty or fewer samples. Routinely, the laboratory includes a matrix duplicate (a sample the laboratory divides into two aliquots) in inorganic test batches and a matrix spike duplicate (a duplicate of the matrix spike) in organic test batches. The type of duplicate to include in a batch is modified based on specific project requirements. A Laboratory Control Standard Duplicate (LCSD) is included if insufficient sample is available to perform a duplicate on a sample. Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). If the RPD is above the control limit, the sample set shall be re-analyzed for the parameter in question or the failure is documented in the case narrative. In inorganic analysis, RPD's are not considered applicable if the concentration in the sample/duplicate is less than 5X the reporting limit.
- 4.4.5 If required by the method, each sample is spiked with the appropriate surrogate standards prior to extraction and analysis. The results of surrogate standard determinations are compared with the true values spiked into the sample matrix. The percent recoveries of the surrogate standards are calculated and reported with the sample results. If recoveries are outside the control limits, corrective action or comment in the case narrative is required. Surrogates are reported as diluted out, in an analysis requiring extraction, if a dilution greater or equal to 10X is performed on the sample. The specific corrective action required is documented in each SOP. Surrogates are used to monitor instrument performance, extraction performance, and matrix affects in each sample analyzed.

- 4.4.6 Internal standards are added to GC/MS Volatiles, GC/MS Semi-volatiles, and select GC methods prior to analysis. A continuous flow internal standard is used for ICP analysis. Internal standards are used to correct for minor variations in retention times and/or response. In most cases internal standards are defined in the method. If not defined an internal standard that is similar in response but not present in the sample (such as deuterated or less common isotopes) shall be used. Internal standard performance is monitored as part of the method performance for response and, if applicable, retention time.
- 4.4.7 An independent calibration verification (ICV) is analyzed following an initial calibration and before any samples are analyzed. The ICV is a standard from a different manufacturer from the initial calibration, if available, or an independent lot if there is only one supplier.

4.5 Statistical Control

As part of the analytical quality control program, the precision and accuracy for each analytical method is established by the use of control charts. The charts are used to assess the method performance over a period of time. A minimum of twenty measurements of precision and accuracy are used to establish a chart. In general, control limits of \pm three standard deviations are utilized. Marginal exceedences shall also be charted using \pm four standard deviations if marginal failures are allowed by the project.

- 4.5.1 Control charts are developed to predict trends (positive or negative) in the analytical processes and to determine when an analysis is out of control. Examples of situations that show up in control charts are:
- Shift in mean - this is usually caused by incorrectly prepared standards or reagents, contamination of sample, problems in instrument calibration, or analyst error.
 - Trend of mean downward - this is usually caused by deterioration of standards or reagents.
 - Trend of mean upward - this is usually caused by concentration of standard due to evaporation of solvent or deterioration of reagents.
 - Increase in variability - this is usually caused by poor technique by the analyst or deviation from procedure.
- 4.5.2 Precision is the measure of how closely multiple analyses of a particular sample agree with each other. To determine the precision of the method and/or laboratory analyst, a routine program of duplicate analyses is performed. The results of the duplicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision. The relative percent deviation (RPD) for duplicate analyses is defined as 100 times the difference (range) of each replicate set, divided by the average value (mean) of the duplicate set.
- 4.5.3 Accuracy is the measure of the closeness of an observed value to the "true" value (theoretical or reference value or population mean). The accuracy of an analytical

method and/or the laboratory analyst is based on the analysis of laboratory control standards. The results laboratory control standards are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R). The %R is defined as 100 times the observed concentration divided by the true concentration of the spike.

- 4.5.4 Uncertainty can be calculated using the Quality Control based Nested approach as described in SOP QA-013. Uncertainty will only be calculated if requested by the client. The approach uses batch QC over a period of time. This approach can also incorporate sample uncertainty if field QC is available. A minimum of twenty measurements is required.

4.6 Audits

Internal audits are scheduled and documented by the QA Department. A member of the QA Department or designee performs the audits as described below. As part of the scheduling, all technologies and Departments must be reviewed at least annually. Audits are documented with records maintained in the QA Department. The goal of internal audits is to determine if lab activities meet the requirements of the quality system and the NELAC standard.

- 4.6.1 System audits are performed to determine if all aspects of the QA program are operational. Through use of a checklist (published NELAC checklist), the QA/QC Director reviews all information pertaining to QA system, summarizes the situation and notes any deficiencies. A report is prepared based on the audit and is distributed to management in a timely manner. The report is also discussed with laboratory personnel so that a concerted effort can be made to correct any deficiencies as well as provide positive feedback. The QA/QC Director reviews the following elements of the program:

- Sample handling, including custody and storage procedures
- Sample analysis
- Records
- Preventive maintenance
- Check sample programs (proficiency testing)
- Training
- Project Management
- Report Generation

- 4.6.2 Method audits are performed throughout the year. These audits focus on a specific method or technology. An analyst is observed performing the method including QC and client samples. The goal of the audit is to determine if the SOP is being followed and traceability of the standards and documentation is maintained. Additionally, the SOP is reviewed for method compliance and is updated as necessary. Part of the system audit is performed at this time including training of analyst performing the tests and performance

in the PT program. After the audit a report is issued to the supervisor. Follow-up audits are necessary to determine if changes are being implemented.

4.6.3 Monthly audits are performed to check support system compliance. A member of QA staff performs these audits and a report is issued to the General Manager and Laboratory Manager. Follow-up and corrective action is performed as necessary. At least the following items are reviewed:

- Calibration of support equipment.
- Temperature checks of ovens, incubators, refrigerators, and freezers are performed daily.
- Logbook documentation and review, and use of strikeout corrections.
- Expiration of standards used.
- Sample chain of custody and tracking of samples.
- Re-Extractions.
- Certificate of analysis.
- Scheduled equipment maintenance is performed.
- Tag-out policy is used.
- Storage blank analysis.
- Safety equipment is being maintained.
- Labeling of Standards and Reagents
- Chain of Custody Procedures

4.6.4 External audits and assessments are scheduled through the QA/QC Director with regulators and clients. It is the policy of GCAL that all information pertaining to that client or scope of accreditation be available for review by the auditor(s). Client confidentiality must be maintained throughout the audit process. The QA/QC Director is responsible for monitoring the audit, reviewing all findings, issuing a corrective action plan in a timely manner, and follow-up to ensure agreed changes are implemented. All documentation pertaining to the audit is kept on file.

4.6.5 Annual Management Review - The management will review the laboratory quality system annually. The review will ensure the suitability and effectiveness of the quality system and introduce any necessary changes and improvements. This is a system wide assessment. After the review, the QA/QC Director shall write a report summarizing the findings and any new policy decisions. The review will include at least the following:

- Matters arising from the previous review
- Suitability of policies and procedures
- Reports from managerial and supervisory personnel
- Outcome of recent internal audits
- Corrective and preventive actions
- Assessments by external bodies
- Results of interlaboratory comparisons or proficiency tests
- Changes in volume and type of work

- Client feedback and complaints
- Staff training needs
- Quality control activities

4.7 Nonconformance/Corrective Actions

A non conformance is any indication or judgment that a product or service has not met the requirements of the relevant specification, contract, or regulation. It is the state of failing to meet the requirements. Corrective action is the action taken to eliminate the causes of an existing non-conformance to prevent recurrence. Nonconformance identification and corrective action are an integral part of GCAL's plan for quality assurance in sample analysis. Every attempt is made by laboratory staff to comply with any requirements set forth in methods, standard operating procedures, GCAL's Quality Assurance Program Plan, and any client or program specific requirements. When non-conformances occur and are not correctable on the spot, the occurrence is documented in the case narrative of the final report and the client is notified on the nonconformance. When errors, deficiencies or out-of-control situations develop, corrective action is initiated. The following nonconformance identification and corrective action programs are used in the laboratory and are described below.

- 4.7.1 Laboratory non-conformances and corrective actions are documented on a Non-Conformance/Corrective Action Form (NCCAF). NCCAF's are tracked by a controlled logbook and maintained by the QA department. Any GCAL employee that identifies a non-conformance shall initiate corrective action by notifying the QA/QC director or laboratory manager, or they can initiate the process by filling out the non-conformance section of a NCCAF and submitting to the QA/QC director. Once initiated, it is the responsibility of the QA/QC director to see that a root cause analysis is performed, a resolution is identified and any follow up action determined necessary is completed. If corrective action is determined to be unsuccessful, the process is repeated. When identified non-conformances result in a change to reported data, a corrected report is prepared, the client is notified of the change, and the corrected report is submitted to the client. The correct report file copy is then attached to the top of the original report file copy.
- 4.7.2 Sample integrity problems determined at login are documented in a Login Discrepancy Form. This form is completed at the time the discrepancy is noted at login and forwarded to the assigned project manager. It is the responsibility of the assigned project manager to contact the client for instructions. These instructions are documented on the form. If the client request to proceed with analysis, this is communicated with login personnel and the samples are released for analysis. If re-sampling will occur the samples are sent to disposal. The original Login Discrepancy Form is kept in the report file copy.
- 4.7.3 On-the-spot or immediate action usually applies to spontaneous, or generally non-recurring problems, such as an instrument malfunction. Any staff member who

detects/suspects nonconformance to previously established criteria or procedure in equipment, instruments, data, methods, etc. shall immediately notify the appropriate department supervisor and/or Laboratory Manager. In many cases, the staff member will be able to correct the problem. Acceptable on-the-spot corrective actions are documented in SOPs and in maintenance logbooks. Examples include re-analysis of a failing LCS, re-extraction, etc. Trends in re-extracts are monitored. When a trend is identified a root cause corrective action is triggered.

- 4.7.4 Prep non-conformances are documented on a Re-extract Form and are logged and tracked by a controlled logbook. Reason for the prep batch re-extraction can include method blank failures, LCS failures, and surrogate failures. Trends in re-extractions are monitored by the QA department. When trends are identified, a corrective action is initiated to determine the root cause using the steps described above in section 4.7.1.
- 4.7.5 If the result of a corrective action or audit cast doubt on the validity of a sample result, the client must be notified. Client notification and further instructions must be documented.

4.8 Customer Inquiries/Complaints

Customer inquiries and complaints are initially received by project managers. If the inquiry requires follow-up action, the project manager fills out a Client Inquiry Form and logs the inquiry into the Client Inquiry Logbook. The inquiry is then sent to the supervisor of the lab or to a member of management for investigation. Following the investigation, a response is recorded on the Client Inquiry Form and the client is notified of the outcome of the investigation. This investigation can include a complete review of the raw data and reanalysis of the sample if applicable. If the inquiry uncovers a lab error and involves further corrective action, the project manager then completes a Corrective Action Form and logs the corrective action into the Corrective Action Logbook. At this point, the corrective action is turned over to the QA/QC department for follow-up and review. If required, the final report is reissued with the appropriate corrections. The report is marked as resubmitted and the reason for the resubmittal is documented in the case narrative.

4.9 Service to Clients

Periodically, GCAL will seek feedback, both positive and negative, from customers in an effort to identify areas where improvement is needed. This feedback can come from audit reports submitted to us by various clients or through questionnaires which are sent out to customers. This feedback is then used to improve our quality system, testing activities, and service to our clientele. In addition, sometimes immediate communication or feedback is need to clarify certain aspects of upcoming projects or to correct encountered problems with current projects.

5 Data Documentation, Validation, and Reporting

Data validation is performed to check data integrity and to verify that the data is correct and of an acceptable quality. Data integrity involves reviewing all documentation for errors and mistakes. It includes review for correct documentation of sample ID's, verification that holding times were met, transcription errors, correct calculations, complete records, and for acceptable chain of custody documentation. A review of the data is performed to verify the results and to assure that all QC is within acceptable criteria. The data is reviewed according to the criteria that applies to the particular analysis and according to the client specific project requirements. The reviewer will identify unacceptable data and initiate the appropriate corrective actions. The documentation of data shall be performed in a manner that allows for the historical reconstruction of results by internal or third party validators.

5.1 Recording data

All raw data is recorded in bound books and/or by instrument printout. This includes calibration, LCS, matrix spikes, duplicates, reagent blanks, calculations, dilutions and any notations concerning a given analysis. If data is recorded by hand, it must be done in ink. It is inappropriate to have pencils, erasers, or correction fluid at the bench.

Data is kept either as a hardcopy, electronically, or both. All data must be protected by the use of audit trails, passwords, and controlled logbooks. If changes or corrections are necessary, it must be performed in a way that maintains the integrity of the data. Changes must be initialed and dated and corrections made using a single line strikeout. If the record does not allow space to clearly show the change, write it in the comments or at the bottom of the page. If electronic files must be changed, the file must be renamed so original information is not lost. If an entire batch of data must be reprocessed all files must be renamed. Reasons for doing so must be written in the comments and/or fully documented using a correction form. At no time shall data be obliterated for any reasons.

Electronic data is backed up and protected by the IT Department. All schedules and procedures are fully documented in IT SOPs.

5.2 Data Reduction

Data reduction includes all activities that convert instrument/computer responses into reportable results. This involves calculations, compound identification, and QC sample calculations. Final results are obtained by direct reading from the instrument or calculations based on instrument readings, output, or responses. Manual data reduction is performed by calculating results with the appropriate formula. Manually entered information such as the sample ID is reviewed for accuracy on the hard copy. Computer data reduction requires that the analyst verify information used in final calculations is entered accurately. The analyst must also review the raw data for properly identified components, possible interferences, confirmation requirements, and acceptable readings for multiple integrations.

5.3 Data Review

5.3.1 All data undergoes an extensive review process. The analyst performs the first level, often as the analysis is being performed. Data is uploaded or manually entered into the LIMS. This review includes the following:

- The calibration meets SOP/project criteria and frequency, and it supports the required detection limit.
- All reagents and standards used within expiration date.
- The method blank pass criteria.
- All applicable QC is performed and meets criteria.
- Dilutions performed as necessary, and the reason documented.
- Is there anything unusual about the sample that is affecting the data? This includes submitted duplicates that are obviously different, presence of interference, analysis past hold time, etc.
- All documentation is complete.

See SOP QA-002 for more details on analyst review.

It is the analyst's responsibility to document any problems and communicate these to the supervisor. If an immediate solution is not found, such as one re-analysis of QC that passes criteria, the problem(s) must be communicated to the Laboratory Manager, Technical Director, QA/QC Director, and/or Project Manager. An Analyst is not authorized to continue with an analysis if the calibration or QC results violate a requirement of the SOP or project. Notify the supervisor immediately and start the corrective action process. Samples shall be held until further documented instructions are received.

5.3.2 The data is then reviewed and validated by the Department Supervisor or designee. Data is validated in the LIMS. This review includes the following:

- The data meets SOP and/or project requirements, including acceptance criteria, frequency of calibrations and QC, and detection limits.
- A set of analyses is logical. For example nitrite is not greater than nitrate + nitrite.
- All calibration/QC failures are clearly described in the batch exception reports. This includes descriptions of allowed failures such as an allowed number of marginal exceedences in the LCS.
- Dilutions are described in the batch exceptions.
- Times and dates are logical and correct.
- Documentation is complete so that the data can be reconstructed based on the information provided.
- Standards and reagents are used appropriately.
- All storage and preservation requirements were met or violations fully described.

- Data is consistent with historical results when available.
- Result is within permit requirements when known.
- Calculations are correct.
- Manual integrations are appropriate and documented.

See SOP QA-002 for more details on secondary supervisor review.

5.4 Data Validation

After the complete package is assembled, the data validation manager or his designee will review the data. This review will include the following at a minimum:

- The correct package has been prepared.
- The package is complete (includes all requested analysis, forms, reports, chain of custody, and raw data as appropriate).
- Project Specific Data Quality Objectives and/or GCAL requirements have been achieved.
- Exceptions and any information that can impact the data are clearly identified in the case narrative.
- Data is flagged appropriately and the data flags are clearly defined.
- Raw data and data reports are consistent.
- All samples have been analyzed and IDs are correct.
- Reporting limits are supported by the calibration.
- Dilution schemes are justified and correct.
- Calculations are correct.

If problems/questions are identified during review, a corrective action form must be completed. The corrective action identifying the problem and the report is sent back to the supervisor(s) for review and correction. Following review and correction, the report is returned to the validator. All steps are documented using the corrective action form. Once complete the corrective action form is returned to QA/QC Department. If the problem is systemic, the corrective action must be forwarded to the QA/QC Director for review and oversight.

5.5 Data Reporting

Hardcopy and electronic reports are the laboratory's product. It is therefore imperative that the report accurately and completely reports results determined by the lab. Any modifications or departures from SOPs are clearly communicated in the report by the use of data qualifiers and the case narrative.

When all requested analysis have been reviewed and validated by the supervisor(s) a preliminary report is sent via e-mail if requested by the client. This report is subject to change if review of the data by report validation indicates a problem. The client shall be contacted about changes in the preliminary report as soon as they are identified and corrected. The final report is then printed. GCAL has the capabilities to produce several levels of reports. These

include a LIMS report with batch QC, a CLP like forms package, a full CLP like deliverable package, and various other formats. The client specifies the level of reporting when the samples are submitted.

After the reports have been authorized and signed by the Validation Manager or designee they are sent through the project manager for release to the client. The QA Director or designee must review a minimum of 10% of all federal program reports, including DOD/AFCEE packages. The QA review is performed following the release of the final report outside of the day-to-day process. The Project Manager is responsible for checking that the report level is correct and the report is signed. After final approval of the reported data, various electronic deliverable formats can be produced to submit data by electronic means. See SOP's LAD-003 and QA-003 for more details on assembling final reports, reviewing final reports, and QA review.

5.5.1 All test reports include the following:

- A cover page that includes a title, "Analytical Results", name, address, and telephone number of the laboratory, work order number, which uniquely identifies the report, and the name and address of the client, contact, and project name, and NELAP certificate number,
- GCAL contact person for questions,
- Pages are numbered sequentially with a total number of pages written in the Laboratory Endorsement page, or numbered as # of total # (for example 1 of 50),
- Signature of Data Validation Manager or designee,
- Statement that the report relates only to the samples reported,
- Statement that the report shall be reproduced only in full and with the written permission of GCAL,
- Case narrative indicating any anomalies, method or QC failures during sample analysis,
- A report sample summary including the sample ID, lab ID, matrix, and collection and receipt date/time.
- The test results that include prep and analytical methods, prep and analysis date and time, prep and analysis batch, weight or volume of sample prepped/analyzed, units, indication of dry weight correction where applicable, results, reporting limits, and data qualifiers,
- QC summary with qualifiers as appropriate, and
- Chain of custody, log-in check sheets, and log-in discrepancy form where applicable.

5.5.2 Additional information shall be provided to clients when requested through reports. This information includes:

- Copies of raw data and logbook entries for submitted analysis,
- Instrument calibration summary and raw data,
- Method detection limits,

- Summaries for surrogate recoveries, internal standards, instrument tune, and method blank summary,
- Manual Integration Summary
- Additional information as requested.
- Identification of sub-contracted work.

5.6 Sub-contracting

Sub-contracting laboratories will be reviewed with an emphasis on their overall quality control practices and compliance to GCAL quality assurance requirements. GCAL bears the responsibility of all sub-contracted work performed by a sub-contractor selected by GCAL. Any laboratory used for subcontracting must be certified or accredited if required for the project and documentation of such must be kept on file. The QA/QC Department or project manager will submit a request to the lab to provide verification of certification or will notify the appropriate accrediting authority to verify certification. Project requirements must be communicated to the sub-contract lab in a timely manner. When data is received it is reviewed by report validation. GCAL will facilitate any client comments or complaints regarding sub-contracted data. The sub-contract laboratory must have successfully completed an assessment by the applicable DOD component (AFCEE, DOD QSM, Army Corp, etc.) for use in all DOD work. The sales representative and project manager verifies compliance with the DOD QSM or other project criteria before the start of the project. Records of compliance must be supplied by the sub-contract lab and kept on file. If testing is subcontracted to another laboratory, the client will be notified in writing.

5.7 Data Storage

The laboratory will retain all records related to sample analysis including raw test data, calculations, derived data, calibrations and copies of test reports. These records are archived in accordance with regulatory requirements for a minimum of ten years or as required by specific client contracts. If the laboratory is going out of business, clients will be notified at least 60 days (if time permits) prior to closure of the laboratory and will receive a final report for all submitted samples. The client notification will request instructions on the retention or distribution of laboratory records and will provide contact information for after the closure. Software/hardware permitting the access of electronic data must be maintained.

The copy of client reports is stored in a room requiring key-card access. All reports must be signed out using the archived reports logbook. Client reports and chain of custodies are also scanned for electronic storage. All archived logbooks, corrective actions, PT results, training records, and other QA/QC reports are stored in a locked storage closet. Only members of the QA/QC Department have access to these records. Written and printed data records (bench sheets, logbooks, electronic printouts, etc.) are scanned before being boxed and placed in storage. Electronic data is stored on a dedicated server. This server is backed-up daily. Approximately 1 year of electronic data is accessible at workstations. Data removed from the servers and stored on tapes can be reloaded by submitting a request to IT. The safety officer

keeps safety and disposal information. The Comptroller in locked files keeps personnel information.

Archived data is stored on-site until capacity is met. The oldest archived data is then moved to a secure storage facility. The storage and on-site facility are monitored and protected from fire and theft. Electronic data storage is free from magnetic sources. It is the goal of GCAL to have redundant copies (hard and electronic) to prevent loss of records due to being misplaced or environmental deterioration or catastrophe.

6 Facility Description and Equipment

6.1 Laboratory Facilities

GCAL is a full service environmental laboratory. The laboratory was established in 1979 with a staff of two and has grown to its present size of over 50 employees operating in a modern laboratory space of 20,000 square feet.

The laboratory's working areas are subdivided into areas for instrumental analysis, wet chemistry and sample preparation. These areas are designed to allow for a safe and comfortable working environment with special attention having been given to ventilation, airflow patterns and environmental controls. Administrative and Marketing areas are located for optimization of supervision and to allow for efficient handling of paperwork and results. The laboratory is protected by an electronic security and fire monitoring system. A floor plan of the facility is included in Appendix D.

6.2 Procurement and Inventory Control

Chemical reagents, solvents, gases, glassware and general chromatographic supplies are ordered as needed to maintain sufficient quantities on hand for use. Purchase orders are maintained as an inventory control of materials ordered by the laboratory. All orders are processed through central receiving and routed to the appropriate departments. Routine supplies are maintained on site in an inventory control stock room.

The purchase of analytical instrumentation is based on anticipated sample volume and the need to maintain superior quality data. Specifications are carefully examined to be sure new instrumentation meets current and anticipated needs. Warranty and service contract information is gathered at the time bids are reviewed and this information is considered in making the final selection. An extensive performance check-out before the instrument is accepted is mandatory. New equipment must undergo a rigorous method validation before being put into production. Operators of new instruments are sent to training courses if necessary.

Inventory records are maintained for all major capital equipment. Major suppliers of consumable items are:

Allometrics	Templet & Templet	Dionex
Fisher Scientific Company	Supelco	CPI
Environmental Express	Perkin-Elmer	Shimadzu

6.3 Capital Equipment

Laboratory equipment and instrumentation are maintained in compliance with instrumentation manuals. All equipment is kept in working condition to allow for conformity to each approved method. The key instrumentation such as Gas Chromatography, Gas Chromatographs/Mass Spectrometers, ICP and Atomic Absorption Spectrometers has maintenance contracts with their

respective suppliers. A list of instrumentation and equipment is maintained by the QA/QC Department and is included in Appendix C.

6.4 Equipment Operation and Calibration

Equipment is defined as any non-disposable mechanical and/or electronic device used in the generation or measurement of data.

- 6.4.1 The calibration of instruments and support equipment is required to ensure that the analytical system is operating correctly and functioning within acceptable precision, accuracy and sensitivity limits. Calibration is defined as the systematic determination of the relationship of the response of the measurement system to a known standard. The calibrations or calibration checks are performed with reference standards traceable to primary standards (e.g. NIST or other certified standards). If traceable chemical standards are not available, standards are prepared according to the laboratory quality control procedures or the project's requirements. The calibration requirements for each type of equipment or instrument are defined in the standard operating procedures. Additionally, specific requirements are defined in a project plan. Table 6-1 summarizes the calibration requirements of the lab.
- 6.4.2 It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results. The inability to achieve calibration is an indication that the equipment needs maintenance. It is not acceptable for an analyst to repeat analysis of calibration or QC standards beyond what is allowed by the SOP until "acceptable" results are achieved.
- 6.4.3 If equipment outside the permanent control of the laboratory is used, it must meet the same criteria. The laboratory shall ensure that the function and calibration status of the equipment is checked and shown to be satisfactory before it is put into service. The equipment must meet all requirements of LADEQ regulations/NELAC standards.

6.5 Equipment Maintenance

Maintenance is defined as cleaning and/or replacing equipment components to assure that the equipment has been properly and periodically serviced and is in satisfactory condition. The equipment manual is a good guideline to determine preventive and routine maintenance schedules. These manuals also assist in identification of commonly needed replacement parts so that an inventory of these parts can be properly maintained.

- 6.5.1 A maintenance log is issued for each piece of equipment. It shall be maintained by the analyst to describe problems, the maintenance performed on the instrument and outcome. This includes routine service checks by laboratory personnel (unless described in the SOP) as well as factory service calls. This log also provides a written source for future use in preventive maintenance. The logs are periodically reviewed by QA.

- 6.5.2 In order to prevent system down time, minimize corrective maintenance cost and to help insure data validity, GCAL uses a system of preventive maintenance. All routine maintenance is performed as recommended by the manufacturer. Maintenance contracts are purchased for most instruments. This insures periodic preventive maintenance visits by factory authorized service representatives and immediate service for corrective actions if required.
- 6.5.3 When a piece of equipment is deemed defective, it is taken out of service and identified with an "OUT OF SERVICE" label. For support equipment such as balances, ovens, coolers, and pipettes, the QA/QC Department is notified so that proper servicing and repair can be scheduled. The analysts perform routine and preventive maintenance for major instrumentation. If outside service is necessary, the Department Supervisor schedules it, with approval from the Laboratory Manager. Satisfactory instrument performance must be verified prior to returning to service any repaired equipment.
- 6.5.4 Table 6-1 is a list of support equipment calibration frequencies. In addition to the stated frequencies, calibrations are performed prior to first use and upon evidence of deterioration. Class "A" glassware is only verified upon evidence of deterioration. Calibration acceptance is based on 10 replicate measurements. See SOP GEN-010 for more details.

Table 6-1 Equipment Calibration

Equipment	Calibration*	Frequency
Analytical Instrument	Traceable standard	Each day of use or as required by instrument manual
Oven and Refrigerator	Calibrated thermometer	Each day of use
Thermometers	NIST Thermometer	Annually (Mercury), Quarterly (Digital)
NIST Thermometer	Certified off-site	As required by certificate
Balance	Certified weights	Each day of use, certified semi-annually
Weights	Certified off-site	As required by certificate
Adjustable pipettes	Weight	Each day of use
Non standard lab ware	Weight	By lot
Non-class A volumetric	Weight	Quarterly
Agitators (TCLP, SPLP)	Stop watch	Monthly

* Acceptance criteria are included in logbook used to document check, or in certificate.

6.6 Reagents

- 6.6.1 All solvents used for preparation of standards must be of acceptable purity to not interfere or invalidate the test. Purity of reagents must meet the reference method requirements and must not invalidate the test as shown by the acceptability of method blanks.
- 6.6.2 Reagents must be stored as specified by the manufacturer, and must be disposed of after the expiration date. If no expiration date is supplied, label acids and bases for five years from receipt, and other reagents as one year from receipt.

- 6.6.3 Neat chemicals must be stored as specified by the manufacturer, and must be disposed of after the expiration date. If no expiration date is supplied, label the neat chemicals for 10 years from receipt.
- 6.6.4 All reagents must be in labeled bottles with the date of receipt and date opened marked in permanent marker.
- 6.6.5 Reagent water is available throughout the lab. GCAL uses de-ionized water supplied by US Filter. The water conductivity is monitored daily and is serviced when necessary.

6.7 Standards

Preparation of standards for calibration or QC must be made from materials of known purity, (98% or better preferred) or from purchased concentrates certified by NIST, EPA, or other acceptable agencies.

- 6.7.1 Stock standards can be kept up to one year if the manufacturer indicates no expiration date. Upon preparation of the standard, the following items must be recorded on the bottle containing the standard: laboratory assigned ID, standard name, concentration, initials of the analyst preparing the standard, date prepared, and expiration date. All other information regarding the standard including solvent used, lot number(s) of solvent used, the analyte source, purity and lot number, expiration date, concentration, dilution procedure, analyst's initials, and date prepared must be entered in the log book.
- 6.7.2 Preparation of intermediate standard solutions is necessary for many tests. These working standards include calibration standards, spiking solutions, surrogate solutions, internal standard solutions, etc., and must be stored as suggested by the manufacturer when not in use. Working standards for the analysis of volatile organic constituents must be prepared at least once in two weeks or more often if required by the method or if performance is compromised. Working standards for the analysis of semi-volatile organic constituents and pesticides are prepared as needed or every six months. Working standards for trace metal analysis is prepared at least once a month for concentrations of 1 mg/L and less. Calibration standards for mercury are digested as needed and calibration standards for graphite furnace are prepared daily. Working standards expiration cannot be longer than the expiration of the parent standard or reagents used. Standard expiration is extended by approval of the QA Director. Acceptable performance must be demonstrated and documentation kept on file. Prepared working standards are verified by comparison to response from the previous calibration as described in SOP GEN-006.
- 6.7.3 The identification of each standard prepared must be unique and all documents related to sample analysis in which the standard was used must contain this unique identification. The documentation shall be such that all of the standard information could be traced from the raw data for the sample.

6.7.4 Freezers and refrigerators are designated for storage of standards. Samples are not stored with standards. Refrigerators or freezers used for storage of standards or samples are monitored for temperature compliance seven days a week. Refrigerators are maintained at $<6^{\circ}\text{C}$ and $>0^{\circ}\text{C}$. Freezers are maintained between -10°C and -20°C .

7 Analytical Methodology

GCAL utilizes methods of analysis that provide evidence of analyte identification, separation from interfering substances, limits of measurement appropriate to that of analyte concentration and reasonable measures of precision and accuracy of the data obtained. Depending upon the analysis requested and the sample matrix, the methods used are official, standard or reference, screening, or modified. Analyses will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. Modification of a method due to sample matrix shall be discussed with and authorized by the client.

If analysis is performed in an alternate manner, the method shall be documented. Documentation is dependent upon the specific instrumentation and data collection and reduction methods used within the lab. Methods used directly from official or standard procedures are referenced as such. Routinely used procedures are available in each department and are also available electronically. Official protocols are used when required or requested.

7.1 Method Validation

Before the performance of methods for reporting to client, each method must be validated. This shall include achieving acceptable calibration and a demonstration of capability. Any work that is performed for government or regulatory purposes shall also have an acceptable limit of detection (LOD) or method detection limit (MDL) study before samples are reported.

7.1.1 Every instrument used to determine results for client samples or QC shall be appropriately calibrated daily before each use. Calibration shall include an initial calibration and continuing calibration as defined in the reference method and described in the SOP. Acceptable performance as defined in the reference method/SOP shall be shown before proceeding with sample analysis. Initial calibrations are verified using an independent standard. Additionally the following shall apply to all calibrations performed:

- Raw data shall be retained to allow reconstruction of the calibration and process to reduce instrument response to concentration. Records shall also include the analyst, date performed, and instrument.
- Samples and QC shall be quantitated using the initial calibration unless the cited method uses alternative procedures.
- The calibration range shall define the working range of the instrument with the exception of metals analysis. For all other analysis the low level standard defines the lowest reporting limit (PQL or LOQ) that is reported to a client. For metals a zero and one point calibration is used. High and low-level checks shall also be included as required by the project or method.
- Sample results exceeding the concentration range (or linear range for metals analysis) shall be diluted.

- The analyst is allowed to drop points out of a calibration curve at the high and/or low ends of the calibration curve if the minimum number of points and the project required detection limits are maintained. Points shall not be removed from the middle of a calibration unless there is a documented reason. The analyst is allowed to re-analyze and replace the suspect point within the same analytical batch or remove the point for all analytes with approval from the QA Director or Laboratory Manager.

7.1.2 Before the implementation of a test method or analyte to a test method, a satisfactory demonstration of method capability is required. This shall include the analysis of four LCS samples with acceptable accuracy and precision. Accuracy and precision is generally defined in the test method. Thereafter, each analyst shall perform a demonstration of capability as part of their initial training and annually. This demonstration shall include acceptable performance in one of the following:

- Acceptable performance of a blind sample;
- A demonstration of method capability; or
- Performance of four consecutive LCS samples with acceptable precision and accuracy.
- If the first three cannot be performed, analysis of samples with results statistically indistinguishable from a trained analyst.

All demonstrations shall be documented on a certification statement and maintained in the analyst's training filed by the QA/QC Department.

7.1.3 A low-level check standard shall be performed at 1-2X the reporting limits. The recovery of this check standard must meet method defined LCS criteria, or lab derived limits if not defined by the method. Alternatively an LOD check shall be performed meeting the criteria of SOP QA-009.

7.1.4 Method detection limit studies shall be performed using 40CFR Part 136 Appendix B. All MDL/LOD are verified using NELAC limit of detection requirements. See SOP QA-009 for a full description of MDL/LOD requirements.

7.1.5 Precision and accuracy of measurement shall be monitored as an ongoing method validation measure. Control charts shall be generated at least annually and control limits updated and compared to method or laboratory historical limits. A copy of the control chart shall be kept on file in the QA/QC Department.

7.2 Methods Outside of Scope of Accreditation

Occasionally a client will request analysis for informational or non-regulatory purposes. Work outside of the scope of NELAC accreditation does not require validation in the same manner as other analysis. Method development will be discussed with the client to meet the client's needs. A letter stating the intent of the work shall be obtained from the client and kept on file.

Reports issued outside of the scope of accreditation shall be identified. This identification shall include either the removal of the LELAP certification number, or, in the case of a mixed report, those methods outside of the scope of accreditation shall be clearly stated in the case narrative.

7.3 Review of New Work

For the laboratory to perform additional work within its scope or to expand its scope of testing a thorough review must be undertaken. Laboratory management considers available resources and current and pending workload prior to accepting new work.

It is the responsibility of the Laboratory Manager, with input from the department supervisors and General Manager, to assess the ability of the laboratory to accept new work.

Before new work is accepted the QA/QC Director must assess the accreditation needs and obtain all necessary certifications.

7.4 Analytical Methods

The analysis performed at GCAL is listed in the following Tables.

Table Organic Tests Performed

Parameter	Method	Reference
Analysis		
Aromatic Volatile Organics	8021B	2
	602	6
Explosives	8330A	2
Organochlorine Pesticides	608	6
	8081B	2
PCBs	8082A	2
TPHG	8015C	2
TPHD	8015C	2
GRO	8015C	2
DRO	8015C	2
ORO	8015C	2
Petroleum Range Organics	FI-Pro	10
Total Petroleum Hydrocarbons	TX1005/TX1006	13,14
EPH	Massachusetts	15
VPH	Massachusetts	16
Organophosphorus Pesticides	8141A	2
Chlorinated Herbicides	8151A	2
Dissolved Gases	RSK175	9
GC/MS Semivolatile Organics	625	6
	8270C, 8270D	2

Parameter	Method	Reference
GC/MS Volatile Organics	624	6
	8260B	2
GC/MS SIM Semivolatile	625	6
	8270C, 8270D	2
HPLC PAH's	8310	2
Solvents	8015C	2
Alcohols	8015C	2
Methanol	94.03/99.01,HAPS	11
Volatile Organics in Ambient Air	TO-15	17
Extractions and Preparations		
TCLP	1311	2
SPLP	1312	2
Separatory Funnel	3510C	2
Liquid/Liquid	3520C	2
Ultrasonic	3550C	2
Waste Dilution	3580A	2
Soxhlet	3540C	2
Purge and Trap	5030B, 5030C	2
Closed System Purge and Trap	5035A	2

Table Inorganic Test Performed

Parameter	Method	Reference
Metals Analysis		
Aluminum -ICP	200.7	7
	6010B, 6010C	2
Antimony -ICP	200.7	7
	6010B, 6010C	2
Arsenic GFAA	206.2	1
	7010	2
ICP	200.7	7
	6010B, 6010C	2
Barium -ICP	200.7	7
	6010B, 6010C	2
Beryllium -ICP	200.7	7
	6010B, 6010C	2
Boron	200.7	7

Parameter	Method	Reference
-ICP	6010B, 6010C	2
Cadmium	200.7	7
-ICP	6010B, 6010C	2
Calcium	200.7	7
-ICP	6010B, 6010C	2
Chromium	200.7	7
-ICP	6010B, 6010C	2
Chromium VI	7196A	2
-Colorimetric	3500Cr D	3
Cobalt	200.7	7
-ICP	6010B, 6010C	2
Copper	200.7	7
-ICP	6010B, 6010C	2
Iron	200.7	7
-ICP	6010B, 6010C	2
Lead	200.7	7
-ICP	6010B, 6010C	2
Magnesium	200.7	7
-ICP	6010B, 6010C	2
Manganese	200.7	7
-ICP	6010B, 6010C	2
Mercury	245.1/245.2	1
CVAA	7471B	2
	7470A	2
Molybdenum	200.7	7
-ICP	6010B, 6010C	2
Nickel	200.7	7
-ICP	6010B, 6010C	2
Potassium	200.7	7
-ICP	6010B, 6010C	2
Selenium		
-GFAA	270.2	1
	7010	2
-ICP	200.7	7
	6010B, 6010C	2
Silver	200.7	7
-ICP	6010B, 6010C	2
Sodium	200.7	7
-ICP	6010B, 6010C	2
Strontium	200.7	7
-ICP	6010B, 6010C	2

Parameter	Method	Reference
Thallium		
-GFAA	279.2	1
	7010	2
-ICP	200.7	7
	6010B, 6010C	2
Tin	200.7	7
-ICP	6010B, 6010C	2
Titanium	200.7	7
-ICP	6010B, 6010C	2
Vanadium	200.7	7
-ICP	6010B, 6010C	2
Zinc	200.7	7
-ICP	6010B, 6010C	2
Zirconium	200.7	7
-ICP	6010B, 6010C	2
Metal Preparation Methods		
Acid Digestion Aqueous and ICP	200.7 3010A	7 2
Acid Digestion Aqueous GFAA	200.9 3020A	1 2
Acid Digestion Solids	3050B	2
Microwave Assisted Acid Digestion Solid and Organic	3051 3052	2
TCLP	1311	2
SPLP	1312	2
Acidity	2310B	3
Alkalinity	2320B	3
Ash	D482	4
BOD/BODC	5210B	3
Bromide	300.0 9056A	1 2
BTU-Heat of Combustion	D240-92	4
Cation Exchange Capacity	9080	2
COD	HACH 8000/8328	5
Corrosivity	1110A 9040B/4500 H B 9045C	2 2 2
Chloride	SM 4500Cl E 300.0 9056A 9251	3 2 2

Parameter	Method	Reference	
Residual Chlorine	4500-CI G	3	
Fecal Coliform	9222D	3	
Color	2120 C	3	
Conductivity	2510B	3	
	9050 A	2	
Corrosivity Toward Steel	1110A	2	
Cyanide	-Free	335.4	1
	-Total	335.4/ 9012A	1 2
		-Amenable to Chlorination	335.4/ 9012A
	Density	2520C	3
	Fluoride	4500F-D/300.0	31
9056A		2	
Hardness Calculation	2340B	3	
Ignitibility	1010A	2	
	1030	2	
% Moisture	SW846 Dry Weight/2540B	2	
Nitrogen	-Ammonia	4500NH ₃ BE	3
		4500NH ₃ BF	3
		4500NH ₃ BE	3
	-Kjeldahl	4500NH ₃ BF	3
		-Nitrate	300.0
	-Nitrite	9056A	2
		353.2	1
	-Total Nitrate Nitrite	300.0	1
		9056A	2
		353.2	1
Oil and Grease	1664A	8	
	9071B	2	
Oxygen, Dissolved	4500 O G	3	
	4500O-C	3	
Paint Filters Liquid Test	9095A	2	
Phenolics	420.1/420.4	1	
	9066	2	
pH	4500-H ⁺ B	3	
	9040B	2	
	9045C	2	

Parameter	Method	Reference
Phosphorus		
-Orthophosphate	4500PBE	3
-Total Phosphorus	365.1	1
Reactivity		
-Cyanide	7.3.3.2	2
-Sulfide	7.3.4.2	2
Silica, Dissolved	4500Si-D	3
Solids		
-Total Dissolved	2540C	3
-Total Suspended	2540D	3
-Total Solids	2540B	3
-Total Volatile Solids	2540E	3
-Volatile Suspended Solids	2540E	3
-Setteable	2540F	3
Specific Gravity	2710F	3
Sulfate	300.0	1
	9038	2
	9056	2
Sulfide	4500S ² D	3
	9034	2
Sulfite	4500 SO ₃ ²⁻ B	3
Surfactants		
-Ionic (MBAS)	5540C	3
-Non-Ionic (CTAS)	5540D	3
Total Organic Carbon (TOC)	5310B	3
	9060	2
Total Organic Halides (TOX)	9020B	2
Turbidity	180.1	1
	2130B	3
Viscosity	D445	4
Perchlorate	314.0	12
Sample Preparation Procedures		
Alkaline Digestion Cr ⁶⁺	3060A	2
Bomb Prep Method for Solid Wastes	5050	2
Distillation Sulfides	9030B	2
SPLP	1312	2

METHOD REFERENCES

- 1) EPA 600 4-79-020, Methods For Chemical Analysis of Water and Wastes, 1983, second printing. Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100)
- 2) EPA SW-846, Test Methods for Evaluation Solid Waste, 3rd Edition, Update I dated 7/92, Update II dated 9/94, Update IIA dated 8/93, Update IIB dated 1/95, Update III dated 12/96, Update IV dated 1/08.
- 3) APHA/AWWA/WPCF, Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992, Online Edition.
- 4) ASTM, American Society for Testing & Materials.
- 5) Hach Company, EPA Approved Procedures for Water and Wastewater, 1986.
- 6) 40 CFR Part 136 Appendix A, Test Procedures for Analysis of Organic Pollutants
- 7) Method 200.7, Determination of Metals and Trace Elements in Water and Wastes By Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, EMMC Version, May 1994.
- 8) EPA-821-R-98-002, USEPA Office of Water Analytical Methods; Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry, February 1999.
- 9) EPA Standard Operating Procedure
- 10) Florida Department of Environmental Protection, Method For Determination of Petroleum Range Organics, FL-PRO, Revision 1, November 1995.
- 11) NCASI Method DI/MEOH-94.03, Methanol in Process Liquids by GC/FID, May 2000 and NCASI Method DI/HAPS-99.01, Selected Haps in Condensates by GC/FID, February 2000.
- 12) NERL, Office of Research and Development, EPA; Method 314.0, Determination of Perchlorate in Drinking Water Using Ion Chromatography, Revision 1.0, November 1999.
- 13) TNRCC; Method 1005, Total Petroleum Hydrocarbons, Revision 03, June1, 2001.
- 14) TNRCC, Method 1006, Characterization of Nc₆ to Nc₃₅ Petroleum Hydrocarbons in Environmental Samples, Draft

- 15) Massachusetts Department of Environmental Protection, Method for the Determination of Extractable Hydrocarbons (EPH), Revision1
- 16) Massachusetts Department of Environmental Protection, Method for the Determination of Volatile Hydrocarbons (VPH)
- 17) EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

8 Sample Custody and Integrity

GCAL utilizes a Laboratory Information Management System (LIMS) that was specifically developed for the needs of environmental laboratories. Horizon[®], was developed by Chemware, Inc., tracks samples and data throughout the laboratory. Results are available from the LIMS in a variety of hard copy formats. Furthermore, web access can be provided to clients who wish to view their data via the World Wide Web. A password security system prevents clients from viewing any data other than their own.

The following is an example of some of the information that is entered into the system:

1. Sample number (unique to this sample)
2. Job number (unique to this job or set of samples)
3. Date received
4. Time received
5. Date analytical results due
6. Sample description
7. Customer's name
8. Customer's address
9. Group number
10. Storage location
11. Notation of any special handling instructions or priority assignments
12. Billing information - purchase orders
13. Analyses requested

GCAL understands that sample integrity is a vital part of Quality Assurance. Samples submitted to the laboratory shall be logged in immediately, or other action taken to preserve integrity of the sample until it can be logged into the system. Any sample that is suspected of being contaminated, improperly stored or preserved, or improperly prepared, shall be reported

to the client immediately. Storage blanks located in the volatiles refrigerators are analyzed every two weeks. Records of these analyses are maintained in the GC and GC/MS Volatiles laboratories. No sample is analyzed if there is a question concerning its integrity.

After the sample analyses are complete and the final report is issued to the client, samples are held for 60 days from receipt before disposal. Samples are held longer per the customer request. GCAL does not accept evidentiary samples.

8.1 Sample Acceptance Policy

Delivery of samples to GCAL shall constitute acceptance by Client of these Terms and Conditions. Until GCAL accepts delivery of samples by notation on a chain of custody document or otherwise in writing, GCAL is not responsible for loss of or damage to samples. GCAL, at its sole discretion, reserves the right to refuse or revoke Acknowledgment of Receipt for any sample due to insufficient sample volume, improper sample container, or risk of handling for any health, safety, environmental, or other reason. GCAL does not accept samples that contain asbestos, biohazards, or radiological materials. Regardless of prior acceptance, GCAL may return samples at its sole discretion if it is determined that the samples may pose a risk in handling, transport or processing, for any health, safety, environmental or other reason. GCAL also reserves the right to return excessive sample volume to the Client, at the Client's expense.

Samples not consumed in testing will normally be retained for a maximum of sixty (60) days before disposal. Samples will be returned to the Client when requested in writing or when they would pose a disposal problem as a hazardous waste as determined by GCAL, at its sole discretion. The cost of returning samples will be invoiced to the Client. GCAL, in its sole discretion, may also agree in writing to retain samples at a monthly storage charge, agreed upon and payable in advance.

If the Client is ordering the work on behalf of another, the Client represents and warrants that the Client is the duly authorized agent for the purpose of ordering and directing said work unless otherwise stated in writing, and accepted by GCAL.

Sample acceptance policy is sent out with bottle orders in an effort to make sample collection personnel aware of GCAL's policy prior to sampling. Sample acceptance policy is also available electronically upon request.

8.2 Chain of Custody

A complete chain of custody is maintained by GCAL. Each sample when submitted to our laboratory is accompanied by a Chain of Custody form. These forms contain pertinent information about the sample including specific analytical requests, sampling notes, sample condition, customer name and address.

Additionally, information concerning the site name, field identification marks, date and time of collection, sampler signature, and preservation data is recorded.

Samples are tagged, preserved if necessary and stored appropriately (i.e. refrigerator, freezer or shelf). Samples to be analyzed for volatile organic compounds are stored in refrigerators located in the volatiles analytical laboratories.

8.3 Internal Chain of Custody

Samples labels include a bar code. All samples must be scanned each time custody of the container is changed. This information is stored in the LIMS, and includes a complete record of the sample custody from receipt to disposal. Information includes the location of the sample, the date and time of each custody transfer, unique initials of each person assuming custody, and a reason for the transfer.

8.4 Custody Transfer

If a sample requires additional work to be performed by a qualified outside laboratory, a chain of custody form is completed and submitted with a representative portion of the sample. A copy of this form is maintained on file along with similar information located in a logbook. The chosen laboratory must sign and date the form upon receipt and return it, along with any unused sample, upon completion of analysis.

8.5 Sample Kits

Occasionally, a customer will request a sampling kit (bottles, vials, etc.) with which to collect samples. Chain of Custody forms are always sent along with the kit to insure proper sample custody. This form is completed at the time of sample collection and is returned with the samples.

8.6 Shipping Requirements

The Department of Transportation (DOT) regulations shall be used for packaging and quantities of shipment. Shipping containers shall be secured using impact strapping material. Copies of the signed Chain of Custody (COC) forms must be delivered with the containers. Any samples being split with another party must be properly labeled, contain a COC, and be packed and shipped according to DOT regulations.

A laboratory file is maintained listing sample kits prepared for clients. It contains the client name, address, form of delivery, preservative (if requested), sample bottle distribution, and analyses to be performed. Additionally, the date the kit is requested, sent and expected arrival date is included, along with any pertinent miscellaneous information.

9 STANDARD OPERATING PROCEDURES

GCAL employs standard procedures for all work performed. These standard procedures insure that work is completed in a professional and timely manner and that all contractual obligations are met.

Standard safety procedures are also part of GCAL Standard Operating Procedures. Confidentiality and security agreements on all work performed are strictly enforced.

Analytical SOPs must incorporate or reference the following topics:

- Identification of test method
- Applicable matrix or matrices
- Detection limit
- Scope of application
- Summary of test method
- Definitions
- Interferences
- Safety
- Equipment and supplies
- Reagents and standards
- Sample collection, preservation, storage, and handling
- Quality control
- Calibration
- Procedure
- Calculations
- Method performance
- Pollution prevention
- Data assessment and acceptance criteria
- Corrective action for out-of-control data
- Handling out-of-control data
- Waste management and
- References
- Tables, Diagrams, Flowcharts, and Validation Data

SOPs are reviewed annually and are the basis for internal method audits. If no changes are made to an SOP during review, an SOP review form is completed and appended to the last page of the original SOP kept on file. See SOP QA-001 for document control procedures.

10 Sample Handling Guidelines

Inorganic and Conventional Parameters

Parameters	Container	Recommended Quantity (mL)	Preservative	Holding Time
Acidity	P,G	100	<6°C	14 days
Alkalinity	P,G	100	<6°C	14 days
Ammonia-N	P,G	500	<6°C, H ₂ SO ₄ to pH <2	28 days
Biochemical Oxygen Demand (BOD)	P,G	1000	<6°C	48 hours
Bromide	P,G	200	None	28 days
Chemical Oxygen Demand (COD)	P,G	100	<6°C H ₂ SO ₄ to pH <2	28 days
Chloride	P,G	200	None	28 days
Chlorine, Residual	P,G	200	None	Immediately
Coliform, Fecal	P,G (sterile)	100	<6°C, Na ₂ S ₂ O ₃	6 hours
Color	P,G	100	<6°C	48 hours
Cyanide	P,G	1000	<6°C, ascorbic acid, NaOH to pH > 12	14 days
Ferrous Iron	P,G	100	2mHCl/100mL	Immediately
Flashpoint	P,G	100	None	Not specified
Fluoride	P	500	None	28 days
Hardness	P,G	100	HNO ₃ to pH < 2	6 months
Nitrogen, Kjeldahl (TKN)	P,G	500	<6°C, H ₂ SO ₄ to pH < 2	28 days
Nitrate-N	P,G	100	<6°C	48 hours
Nitrite-N	P,G	100	<6°C	48 hours
Nitrate-Nitrite as N	P,G	200	<6°C, H ₂ SO ₄ to pH < 2	28 days
Oil and Grease	G	1000	<6°C, H ₂ SO ₄ or HCl to pH < 2	28 days
Phenols	P,G	1000	<6°C, H ₂ SO ₄ to pH < 2	28 days
Phosphorus, Total	P,G	200	<6°C H ₂ SO ₄ to pH < 2	28 days
Phosphorus, Ortho	P,G	200	<6°C	48 hours
pH	P,G	100	None	Immediately
Radiochemistry				
Alpha, Beta, Radium	P,G	2000	HNO ₃ to pH < 2	6 months
Tritium	P,G	100	None	6 months
Radon, I-131	P,G	1000	HNO ₃ to pH < 2	14 days
Reactivity	G	100g	<6°C	Not Specified
Silica	P, PFTE, Quartz	100	<6°C	28 days
Solids, Dissolved (TDS)	P,G	100	<6°C	7 days
Solids, Suspended (TSS)	P,G	500	<6°C	7 days
Solids, Volatile (TVS)	P,G	100	<6°C	7 days
Solids, Total (TS)	P,G	100	<6°C	7 days

Inorganic and Conventional Parameters

Parameters	Container	Recommended Quantity (mL)#	Preservative#	Holding Time**
Specific Conductance	P,G	100	<6°C	28 days
Specific Gravity	P,G	100	<6°C	28 days
Sulfate	P,G	200	<6°C	28 days
Sulfide	P,G	500	<6°C, Zn acetate, NaOH to pH > 9	7 days
Sulfite	P,G	200	None	Immediately
Surfactants (MBAS)	P,G	250	<6°C	48 hours
Total Organic Carbon (TOC)	P,G	100	<6°C, HCl to pH < 2	28 days
Total Organic Halogens (TOX)	G-TLC (amber)	100	<6°C, H ₂ SO ₄ to pH < 2	28 days
Total Petroleum Hydrocarbon (TPH)	G-TLC	1000	<6°C, H ₂ SO ₄ or HCl to pH < 2	28 days
Turbidity	P,G	100	<6°C	48 hours
Viscosity	P,G	500	None	Not Specified

#Solid and waste samples: Quantity 1-100g, preservative <6°C.

**Holding time for solids and samples is not defined

Organic Nitrogen = TKN – Ammonia-N

Metals

Parameters	Container	Recommended Quantity (mL)	Preservative	Holding Time
Total	P,G	500	HNO ₃ to pH < 2	6 months
Dissolved	P,G	500	Filter on site HNO ₃ to pH < 2	6 months
Solid				
Total	P,G	100g	<6°C	6 months
Hexavalent Chromium:				
Aqueous	P,G	500	<6°C	24 hours
Solid	P,G	100g	<6°C	30/7 days
Mercury:				
Aqueous				
Total	P,G	500	HNO ₃ to pH < 2	28 days
Dissolved	P,G	500	Filter on site HNO ₃ to pH < 2	28 days
Solid				
Total	P,G	100g	<6°C	28 days

CrIII=Total Cr-Hexavalent Cr

Organic Parameters**Volatile Organics**

Sample Matrix	Container	Minimum Quantity	Preservative	Holding Time
Concentrated Waste Samples	G-TLC or G-TLS	2 x 40mL vials or 2-oz wide mouth	<6°C	14 days
Aqueous Samples	G-TLS	2 x 40mL vials	<6°C, HCl to pH < 2,	14 days, 7 days if not

			Na ₂ S ₂ O ₃ if residual chlorine present	acid preserved
Solid Samples	G-TLS or G-TLC	2-oz wide mouth and/or 3 Encores	<6°C	14 days **

**Solid samples collected in EnCore™ samplers must be transferred to a soil sample vial within 48 hours.

Semivolatile Organics, Pesticides/PCBs, Herbicides, PAH's, Petroleum Hydrocarbons

Sample Matrix	Container	Minimum Quantity	Preservative	Holding Time
Concentrated Waste Sample	G-TLC (Amber)	1 Liter	None	14 days until extraction, 40 days after extraction
Aqueous Samples	G-TLC (Amber)	2 x 1 Liter	<6°C	7 days until extraction, 40 days after extraction
Solid Samples	G-TLC	8 oz.	<6°C	14 days until extraction, 40 days after extraction

Parameter	Container	Recommended Quantity	Preservative	Holding Time
Dioxins and Furans**	G-TLC(Amber)	2 x 1 Liter	<6°C	30 days until extraction, 45 days after extraction

**Concentrated wastes and soil samples are collected in 2 oz. to 1 Liter amber glass jars with TLC.

***1005/1006, Petroleum Hydrocarbons -14 days after extraction

TCLP/SPLP Parameters

Parameters	Holding Time from Collection to TCLP Extraction (days)	Holding Time from TCLP Extraction to Preparative Extraction (days)	Holding Time from TCLP/Preparative Extraction to Analysis (days)	Total Time
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals	180	NA	180	360

Reference: 40CFR Part 136 Tables IA, IB, IC, ID & IE and Table II., SW846 Table 4-1 and Table 3-1, SW846 Method 1311 8.5,

Acronym Definitions: (Teflon is a registered trademark of E.I. DuPont)

CLP: EPA Contract Laboratory Program

G-TLC: Glass with Teflon®-lined cap

NA: Not Applicable

G: Glass

G-TLS: Glass with Teflon®-lined septum

P: Polyethylene

10.1 Waste Collection and Storage

Samples are stored in the appropriate cooler for 60 days after receipt. After 60 days, samples are moved to a waste area. The samples are scanned out for disposal on the LIMS. The samples are then stored in the waste staging area until disposal into appropriate drums. Hazardous samples are returned to the client whenever possible to be disposed of with larger quantities of the sample material. Laboratory waste is segregated by laboratory personnel into waste streams, which have been established by the Regulatory Compliance Officer. The waste streams are determined by analysis of the waste and through process knowledge. All laboratory wastes are disposed of in the proper container. No waste is placed in regular trash containers or poured down the drain. Waste is stored in drums in satellite accumulation areas and then in the central accumulation facility. Waste disposal service is provided by approved vendors who will incinerate, landfill, treat, or reclaim the waste based on the characteristics.

10.2 Pollution Prevention

Environmental concerns, risks to employees and the public, and high disposal costs have increased the need and effort of the laboratory to minimize or prevent waste generation. The quantity of chemicals and standards purchased is based on expected usage during its shelf life and the disposal cost of the unused material. The volume of standards and reagents prepared in the laboratory reflect stability and anticipated usage. If possible, methods requiring the use of hazardous chemicals or that produce hazardous waste are replaced with an alternative method. Sample containers are selected based on the minimum volume that is necessary to perform a test, therefore reducing sample waste. Sample sizes are reduced in some cases, therefore reducing the quantities of extraction solvents and reagents.

11 Safety Procedures

GCAL has a comprehensive safety program outlined for all employees. A safety manual is distributed to each employee followed by a training seminar to familiarize the employee with the safety procedures at GCAL.

11.1 Basic Safety Rules

1. All injuries are promptly reported to a supervisor.
2. All hazards are promptly reported to a supervisor.
3. Running and horseplay are not permitted in the laboratory.
4. Smoking is not permitted in the laboratory.
5. Laboratory glassware is not to be used for eating or drinking.
6. Laboratory reagents such as sucrose or sodium chloride shall not be used for food.
7. Eating on the premises is confined to designated areas.

11.2 Arrangement of Furniture And Equipment

Furniture is arranged for maximum use of available space while providing working conditions that are efficient and safe.

Aisles are kept at least 4 feet wide to provide for safe passage of personnel and equipment, and are kept free of obstructions.

Stepladders or footstools are supplied for reaching high objects and are kept out of the way when not in use.

Eyewash stations, safety showers and fire extinguishers are located centrally and care is taken to avoid blocking access to them.

11.3 Hoods And Ventilation

Adequate hood facilities are installed and used where toxic or flammable materials are used. Hood windows provide physical protection and greater control of fumes.

11.4 Spills

Spilled materials are cleaned up promptly. All spills shall be handled as if corrosive or dangerous unless definitely known to be harmless. Spill Kits are located in the laboratory.

Corrosive or toxic materials are not placed in waste cans in the laboratory. When in doubt a supervisor is consulted.

Broken glass is swept up immediately and discarded so as to avoid any injury or cuts.

11.5 Emergency Equipment

Fire extinguishers are located in each room of the laboratory. The paths to these are kept free and clear at all times.

An extinguisher that has been used shall not be returned to its holder until it has been recharged and checked.

Any fire that appears to be too large to extinguish immediately is reported to the fire department at once. All fires, regardless of size are to be reported to a supervisor. Causes shall be determined and necessary steps to prevent a similar accident shall be taken.

Eyewashes are located in the laboratories for irrigation of the eyes if corrosive liquids shall be splashed into them. Tubing attached to faucets in the sink shall also be used to wash the eyes if necessary.

Safety showers are centrally located throughout the laboratory and are used whenever corrosive materials are spilled on an analysts' skin or clothing.

All safety equipment is periodically checked to be sure everything is in working order and is easily accessible.

General first aid kits are located throughout the laboratory. These kits contain first aid products for the treatment of minor cuts and bruises, burns or abrasions, and personal discomfort.

11.6 Protective Equipment

Lab coats and aprons are supplied for all employees of GCAL. Protective clothing is always available to prevent damage to clothing and persons.

Shoes must be worn at all times and must be closed-toe; high heels or sandals are not acceptable.

Eye Protection is mandatory for all personnel working in the laboratory. Safety glasses or goggles shall be worn by analysts to protect the full eye area in designated areas.

Various types of gloves are provided for employees: Insulated gloves are provided for use when handling hot or cold items; Heavy rubber gloves are to be used when handling corrosive liquids or unknown substances; Lightweight disposable gloves are provided for use with toxic or irritating substances.

Air purifying respirators are available for use when working with organic vapors and/or acid fumes for qualified trained analysts. These respirators shall be worn whenever contact with irritating concentrations of these fumes is encountered.

11.7 Storage of Laboratory Materials

All chemicals, reagents and glassware are stored in such a manner that they are easily located and do not present a danger. Heavy items are kept near the floor.

Flammable solvents are stored in special cabinets or in solvent bunker. Only quantities required for immediate use are stored in analytical areas.

Reagents are grouped to prevent danger from hazardous combinations. Acids and bases are stored separately.

Compressed gases are stored away from heat and open flames. Chains or belts to prevent rolling or toppling always contain them. A special cart is used to transport replacement cylinders and empties.

11.8 Chemical And Sample Handling

If there are questions about proper chemical handling the MSDS (Material Safety Data Sheet) is used as reference.

Samples are always treated as if they were hazardous chemicals.

Rubber pipette bulbs are used.

Procedures that produce flames or toxic vapors are performed under a hood.

Chemicals are returned to their proper storage area after use.

All prepared solutions are properly labeled.

Acids are always poured into water when diluting.

Large amounts of alkali are never added to water at one time.

Glass-stopper containers are not used for storing alkaline solutions.

Labels for Acid and Caustic solutions will note the concentrations.

12 Confidentiality

GCAL understands that it must retain in confidence all information obtained through the analysis of samples or the information disclosed to GCAL in order to adequately perform and interpret analyses.

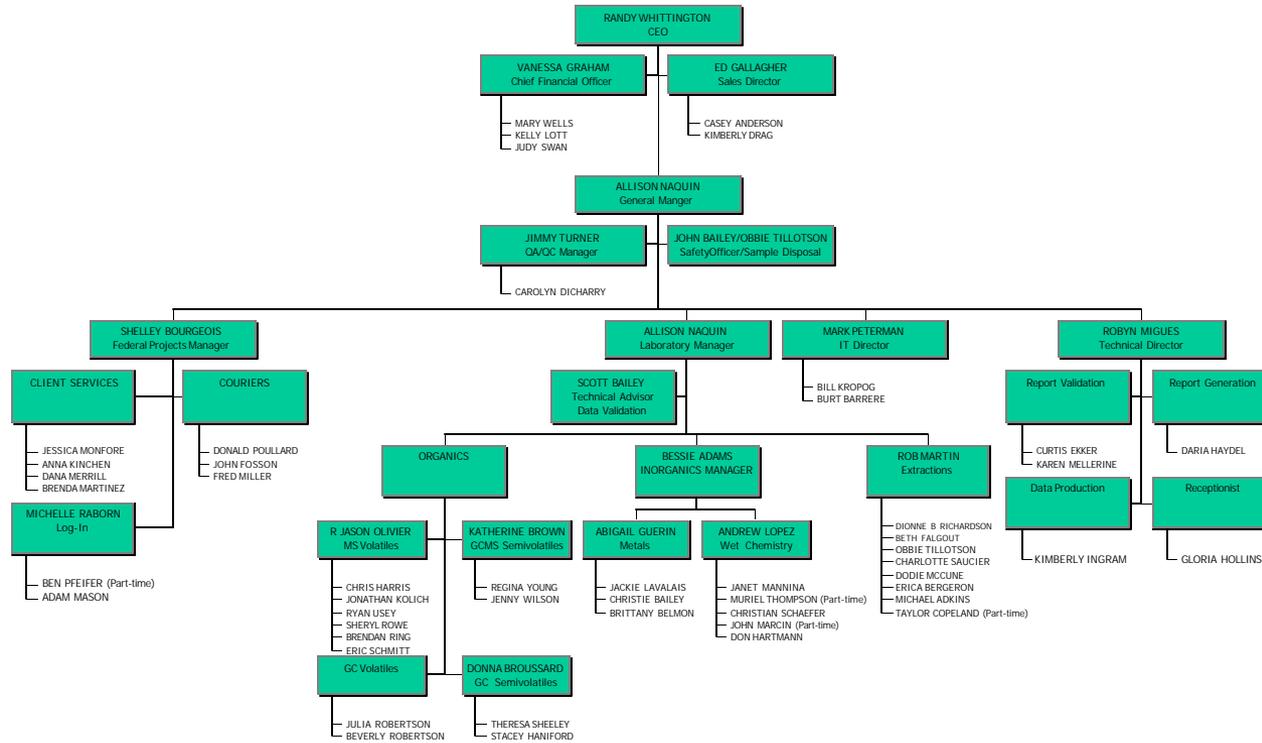
GCAL will maintain the secrecy and confidentiality of any proprietary information it receives or generates.

Only the party who requested the analytical work or consultation, and who will receive the final report (and invoice) will be informed of any findings.

The contracting party will not disclose results of analyses to anyone other than the contracting party without unequivocal authorization.

Appendix A

ORGANIZATIONAL CHART



Appendix B

RANDY K. WHITTINGTON

Current Position:

CEO, Gulf Coast Analytical Laboratories, Baton Rouge, LA, October 2009 - present

Responsible for financial and marketing functions. Responsible for long range planning and structuring of future business operations..

Previous Experience:

Technical Services Manager, Gulf Coast Analytical Laboratories, Baton Rouge, LA, January 1997 – April 2007

Responsible for the management and supervision of Sample Management, Project Management, and Report Generation. Duties include implementing systems for increased productivity in all three sections. Also coordinates communication among these departments and other areas of the laboratory and marketing.

Technical Services Manager, ITS-Environmental Laboratories, Baton Rouge, LA, October 1996 - January 1997

Responsible for the management and supervision of Sample Management, Client Services, and Report Generation. Duties include implementing systems for increased productivity in all three sections. Also coordinates communication among these departments and other areas of the laboratory and marketing.

Project Manager and Data Validation Manager, Terra Consulting Group, Baton Rouge, LA, 1993 - 1996

Performed organic data validation for CLP and RCRA data packages for pesticides, PCBs, volatile and semi-volatile analytical fractions. Responsible for the design and implementation of the analytical aspects needed to generate legally defensible data for a Remedial Feasibility Investigation (RFI) at various large chemical plants. Ensured data validation issues were addressed in the day-to-day operations of the investigation.

Gas Chromatography Supervisor, West-Paine Laboratories, Baton Rouge, LA, 1991-1993

Directly responsible for the supervision of the organics laboratory in Randy

environmental and hazardous waste matrices following current SW-846, 500 and 600 series methodologies. Responsibilities include coordinating and managing of QA/QC for all Gas Chromatography data from sample log-in, extraction, analysis, review and preparation of computerized reports.

Gas Chromatography Laboratory Manager, ETC/Toxicon, Baton Rouge, LA, 1987-1991

Supervised the Gas Chromatography laboratory in the analysis of Organochlorine and Organophosphorus Pesticides, PCBs, Herbicides, PNAs, VOA and Semi-VOAs; supervised all aspects of the GC laboratory including analysis, data interpretation, report preparation, instrument maintenance, method development, and problem solving. In 1990 temporarily relocated to Edison, New Jersey to restructure the Gas Chromatography division while also implementing USEPA CLP and Finnigan QA Formaster; maintained efficiency of twenty-two various Gas Chromatographs.

Education:

BS, Environmental Engineering , Columbia Southern University

Inchcape Managerial Training Skills Workshop - 1993

Finnigan QA Formaster Training

Restek Chromatography Class

Bank One Managing and Financing Independent Business - 16 Hours -
October 1998

ALLISON M. NAQUIN, Ph.D.

Current Position: **Laboratory Manager, GCAL Inc, Baton Rouge, LA, Jan 2010 - Present**
Responsible for coordinating the overall activities of the analytical laboratories on a daily basis and providing long-term direction. Responsibilities include monitoring the scheduling of analytical testing and releasing testing data and results. Continue General Manager duties as described below.

General Manager, QA/QC Manager, GCAL, Inc., Baton Rouge, LA, August 2007 - Present
Responsible for all operations within the facility including laboratory and administrative policies and procedures.

**Previous
Experience**

QA/QC Manager, GCAL Inc., Baton Rouge, LA, April 2005 – August 2007

Responsible for implementing and monitoring the laboratory Quality Assurance Program Plan, conducting internal audits, investigating problem areas, control-chart generation, establishing data-quality criteria, verifying corrective actions are being taken when necessary, directing participation in accreditation programs, and monitoring performance evaluation studies. Additional duties include administering the ethics training/data integrity program and providing reports concerning QA matters to management.

Laboratory Auditor, GCAL Inc., Baton Rouge, LA, February 2005 – April 2005

Support QA/QC functions and perform internal audits. Responsibilities include performing internal audits of lab and writing audit reports. Assist in writing standard operating procedures.

Environmental Scientist Supervisor, Louisiana DEQ, Baton Rouge, LA, December 2004 – February 2005

Served as the Technical Advisor for the laboratory to the Assistant Secretary of the Office of Environmental Assessment. Responsibilities include draft/review and approval of Quality System documents; advise laboratory on technical and quality issues to obtain NELAP accreditation, and audit laboratory activities to NELAC standards. Continue assistance to Lab Accreditation Program.

Environmental Scientist III, Louisiana DEQ, Lab Accreditation Program, Baton Rouge, LA, April 2002 – December 2004

Assess commercial environmental and industrial laboratories to NELAC/ISO standards, and assess quality documents. Responsibilities also include serving as organic specialist to accreditation group, review data packages, assist in training seminars to environmental community, and assists Executive Staff with technical issues.

Environmental Scientist I-II, Louisiana DEQ, Laboratory Services Division, Baton Rouge, LA, February 2001 – April 2002

Responsibilities include analysis of volatile samples by GC/MS, interpretation and reporting data, preparation of data packages, and draft standard operating procedures. Cross-trained on semi-volatile analysis by GC/MS.

Research Assistant, Louisiana State University, Chemistry Department Baton Rouge, LA, August 1998 – December 2000

Responsibilities include directing daily activities of research lab, conduct environmental research, maintain and repair laboratory equipment, and mentor undergraduate students. Also, prepared and delivered seminars on personnel research and related literature, and was liaison between LSU and Southern University for research project.

Teaching Assistant, Louisiana State University, Chemistry Department Baton Rouge, LA, January 1995 – August 1998

Responsibilities include instructor for general chemistry laboratory, tutor undergraduate students, and provides class reviews and exam proctoring for professors.

Independent Contractor, Baton Rouge, LA, September 1998 – November 1998

Performed metals digestion and ICP/MS analysis of environmental samples from an EPA clean-up site. Issued reports to Dr. James Wharton of LSU Chemistry Department.

Adjunct Chemistry Instructor, Louisiana State University, Chemistry Department, Baton Rouge, LA, August 1994 – December 1994

Responsibilities include instructor for general chemistry laboratory, provides instruction in class materials, and administers and grades class-work and exams.

Education

Doctor of Philosophy, Chemistry, Louisiana State University, Baton Rouge, LA, May 2001

BS Chemistry, Louisiana State University, Baton Rouge, LA August 1994

Accreditation Process, Laboratory Ethics, How to Write Quality Documents Training Course, Analytical Excellence, April 2004

Assessments for ISO/IEC 17025 & NELAC (ASI Course 300), Advanced Systems, Inc., July 2003

Data Assessment Training, Analytical Excellence, Inc., May 2003

QA/QC Workshop, Advanced Systems, Inc., May 2003

Calibration and Manual Integration, Analytical Excellence, Inc., May 2003

HAZWOPER 40-hour Training Course, July 2002 and yearly refreshers August 2003 and August 2004

Data Integrity Seminar – Ethics Training, Analytical Excellence, Inc., March 2002 and yearly refresher

Environmental GC/MS Instrument and ChemStation Operation, Agilent Technologies, October 2001

Comprehensive Public Training Program

SHELLEY BOURGEOIS

Current Position:

Client Services Manager, Gulf Coast Analytical Laboratories, Baton Rouge, LA, April 2007 - present

Responsible for the management and supervision of Sample Management and Project Management. Duties include implementing systems for increased productivity and coordinating communication among these departments and other areas of the laboratory.

Previous Experience:

Project Chemist, Conestoga-Rovers & Associates, Baton Rouge, LA, May 2004 - April 2007

Responsible for contracting analytical laboratory services and for QA/QC verification of data. Performed data validations and ensured data validation issues were addressed in the day-to-day operations of the investigation.

Inorganics Manager, Gulf Coast Analytical Laboratories, Baton Rouge, LA, May 1998 - May 2004

Responsible for the management and supervision of the Metals Laboratory. Duties include the management and training of personnel, scheduling of sample workloads, supervision of metals sample preparation, analysis of samples by various analytical instrumentation, coordination of laboratory QA/QC projects, and maintenance of procedures to QA/QC guidelines. In addition, responsibilities include comprehensive data review and validation for the laboratory as well as the coordination of higher level QA data packages.

Metals Analyst, Gulf Coast Analytical Laboratories, Baton Rouge, LA, December 1997 - May 1998

Responsible for analysis of samples by various instruments including GFAA, Flame AA, ICP and the mercury analyzer. Additional responsibilities included data reduction and posting in the LIMS.

Head Technician, American Radiation Services, Baton Rouge, LA,
November 1996 - December 1997

Responsible for coordinating sample analysis and field services.
Supervised sample receipt, preparation, analysis, and report generation.

Analyst, ITS Environmental Laboratories, Baton Rouge, LA, July 1996 -
November 1996

Education:

BS/Microbiology - Louisiana State University, Baton Rouge, LA,
December 1995

Perkin Elmer Atomic Spectroscopy Workshop, Baton Rouge, LA, April
1998

Perkin Elmer Optima Instrument Series ICP training, Atlanta, GA, June
1998

ROBYN B. MIGUES

Current Position:

Technical Director, Gulf Coast Analytical Laboratories Baton Rouge, LA, April 2005 – Present

Responsible for report validation and review. Responsible for review of Quality Assurance Project Plans on incoming projects and implementation of such plans throughout the laboratory. Assists the lab in method implementation and development. Additional duties include advising the laboratory on reference methods and improving method performance.

Previous Experience:

QA/QC Manager, Gulf Coast Analytical Laboratories Baton Rouge, LA, January 1997 – April 2005

Responsible for implementing and monitoring the laboratory Quality Assurance Program Plan, conducting internal audits, reviewing reports, investigating problem areas, control-chart generation, establishing data-quality criteria, verifying corrective actions are being taken when necessary, and monitoring performance evaluation studies. Additional duties include providing reports concerning QA matters to management.

**QA/QC Manager, ITS- Environmental Laboratories, Baton Rouge, LA
October 1994 - January 1997**

Responsible for implementing and monitoring the laboratory Quality Assurance Program Plan, conducting internal audits, reviewing reports, investigating problem areas, control-chart generation, establishing data-quality criteria, verifying corrective actions are being taken when necessary, and monitoring performance evaluation studies. Additional duties include providing reports concerning QA matters to management.

**General Chemistry Supervisor, ITS- Environmental Laboratories,
Baton Rouge, LA, June 1994 - October 1994**

Responsibility includes the management and training of personnel conducting inorganic analysis using EPA methodologies. Duties include data validation, QC review, instrument maintenance and method set up.

**Metals Supervisor, ITS –Environmental Laboratories, Baton Rouge,
LA, October 1993 - June 1994**

Responsible for the management and supervision of the Metals section which includes supervision of metals sample preparation, supervision and training of analysts, scheduling sample workload, analysis of samples by various analytical instrumentation and reviewing and validating all data.

**Research Associate, Louisiana State University, Agronomy
Department, Baton Rouge, LA, September 1990 - March 1993**

Prepared and analyzed samples by ICP, maintained ICP and other laboratory equipment, assisted associate Professor of soil and environmental chemistry with laboratory courses and research projects and supervised student workers. Computer experience includes Quattro Pro and Wordperfect.

**Previous
Experience:**

**Spectroscopy and Water Departments Supervisor, James Laboratories,
Lafayette, LA, February 1987 - September 1990
Laboratory Technician**

Prepared and analyzed samples by ICP, Flame Atomic Absorption & Emission, Mercury Hydride System and Graphite Furnace. Performed quality control coordination, trained laboratory technicians, maintained equipment . Prepared and analyzed various sample types.

Education:

BS Geology, University of Southwestern Louisiana, Lafayette, LA, May 1985.

Member - American Society for Quality Control

Perkin Elmer Spectroscopy training course - 1987

Basic Statistics - Pittsburgh Conference Continuing Education Program -
March 1995

Quality Management/Quality Assurance in Industry and in the Laboratory -
ACS Short Course - March 1995

Inchcape Managerial Training Skills Workshop - 1994

Inchcape Testing Services - Environmental Laboratories, Baton Rouge,
Manager and Supervisor Training Retreat - June 1996

Executrain Microsoft Excel 5.0 Beginning For Windows - July, 1996

ERTCO - Thermometer Calibration per ISO - October 1997

Assuring Ethical Practices in The Environmental Laboratory, A Training
Short Course - Analytical Excellence - October 27, 2000

Member - LADEQ Laboratory Accreditation Task Force

JAMES D. TURNER

**Current
Position:**

QA/QC Manager, GCAL Inc., Baton Rouge, LA, May 2008 - Present

Responsible for implementing and monitoring the laboratory Quality Assurance Program Plan, conducting internal audits, investigating problem areas, control-chart generation, establishing data-quality criteria, verifying corrective actions are being taken when necessary, directing participation in accreditation programs, and monitoring performance evaluation studies. Additional duties include administering the ethics training/data integrity program and providing reports concerning QA matters to management.

**Previous
Experience:**

Project Manager, Gulf Coast Analytical Laboratories, Baton Rouge, LA, October, 2004 - May 2008

Responsible for management of client projects and project management activities within the laboratory. Served as the interface between client and laboratory management to achieve client satisfaction with delivery of analytical results on schedule and to the requested level of quality.

**QC Lab Manager, The Wright Group, Inc., Crowley, LA
March 2004 - October 2004**

Responsible for the management of a nutraceutical laboratory for a food fortification company.

Organics Manager, Gulf Coast Analytical Laboratories, Baton Rouge, LA, June 2001- February 2004

Responsible for the management of the GC, GCMS, and the extraction departments of the laboratory.

Training Officer, Gulf Coast Analytical Laboratories, Baton Rouge, LA, January 2000 - 2001

Responsibilities include development of a company training program that will efficiently train employees to ensure compliance with the internal SOP's and the analytical methods. The officer will be responsible for initial training of new employees as well as ongoing training for all employees. The officer will also maintain the training records and the analyst certification program.

**General Chemistry Supervisor, Gulf Coast Analytical Laboratories,
Baton Rouge, LA, January 1997 - Present**

Responsible for the supervision and training of personnel, conducting inorganic analysis using EPA methodologies, correlation and validation of data, maintenance of Standard Operating Procedures (SOPs) and instrumentation, method set up and day-to-day management of the general chemistry laboratory.

**General Chemistry Department Group Leader, ITS -Environmental
Laboratories, Baton Rouge, LA, August 1996 - January 1997**

Responsible for analyst training, instrument maintenance, and scheduling daily work loads.

**General Chemistry Laboratory Technician, ITS -Environmental
Laboratories, Baton Rouge, LA, November 1992 - August 1996**

Responsible for preparation and analysis of standards and samples for most wet chemistry tests. Experience on instrumentation includes Lachat Quick Chem Analyzer, TOC, TOX, HACH Spectrophotometer, IC, HPLC, GCMS, and GPC. Additional responsibilities included initial review of data and associated QC and entry into the LIMS.

**Chemist, La-Mar-Ka Chemical, Baton Rouge, LA, November 1991 -
November 1992**

Responsible for preparation and standardization of chemical solutions. Instrumentation experience included D.L. 40 Auto Titrator and Moisture Analyzer.

**General Chemistry Laboratory Technician, Enviromed Laboratories,
Baton Rouge, LA, June 1988 - November 1991**

Experience includes preparation and analysis of samples by wet chemistry methods and preparation of samples by organic extraction procedures. Introduced to GC/MS. Assisted with sample collection and waste disposal.

Education:

BS, Microbiology, Louisiana State University, Baton Rouge, LA, May 1999
Minor-Chemistry

OSHA 40 hour Hazardous Waste Training Course - August 1991

ITS - Environmental Laboratories, Laboratory Skills Training Program -
August 1995

ITS - Environmental Laboratories, Basic Gas Chromatography Theory - May 1996

Appendix C

GCAL Equipment List

ORG/UNCS	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
GCMSSV 4	MSSV Lab	September-05	September-08	new	AGILENT 5975	US52430653
	MSSV Lab	September-05	September-08	new	AGILENT 6890N	CN10532032
GCMSSV 5	MSSV Lab	November-05	November-05	new	AGILENT 5975	US53931245
	MSSV Lab	November-05	November-05	new	AGILENT 6890N	CN10539069
GCMSSV 6	MSSV Lab	July-07	July-07	new	Agilent 7890	CN10717068
	MSSV Lab	July-07	July-07	new	Agilent 5975C	US71235850
GCMSSV 0	MSV lab	September-01	September-01	new	HP 5890 SERIES II	3336A58851
	MSV lab	September-01	September-01	new	HP 5972	3501A02325
	MSV lab	September-01	September-01	new	Teledyne/Tekmar-XPT	US05279001
	MSV lab	September-01	September-01	new	T/D Solatek 72	USO2294002 (GCAL# 0337)
GCMSSV 5	MSV lab	October-03	October-03	new	HP 5890 SERIES II	3310A48460
	MSV lab	October-03	October-03	new	HP 5971	3307A00395
	MSV lab	October-03	October-03	new	Tekmar LCS 2000	90211015/93154002/9115009
GCMSSV 8	MSV Lab	October-01	October-01	new	AGILENT 5973	US10441235
	MSV Lab	October-01	October-01	new	AGILENT 6890N	US10134037
	MSV Lab	October-01	October-01	new	Teledyne/Tekmar-XPT	US03240004
	MSV Lab	October-01	October-01	new	T/D Solatek 72	US05283001
GCMSSV 9	MSV lab	April-07	April-07	new	AGILENT 5979B	US63234781
	MSV lab	April-07	April-07	new	AGILENT 6890N	CN10647134
	MSV lab	April-07	April-07	new	Teledyne/Tekmar-XPT	US06296004
	MSV lab	April-07	April-07	new	T/D Solatek 72	US07022004
GCMSSV 11	MSV Lab	April-04	April-08	new	AGILENT 5973	US33220204
	MSV Lab	April-04	April-08	new	AGILENT 6890N	CN10407013
	MSV Lab	July-07	July-07	new	Teledyne/Tekmar-XPT	US03140007
	MSV Lab	July-07	July-07	new	T/D Solatek 72	US02098018
GCMSSV 12	MSV Lab	June-10	June-10	new	AGILENT 5973	US10441235
	MSV Lab	June-10	June-10	new	AGILENT 7890A	CN10211053
	MSV Lab	June-10	June-10	new	Teledyne/Tekmar-XPT	US10160001
	MSV Lab	June-10	June-10	new	T/D Solatek 72	US05283001
FUME HOOD	MSV Lab	January-97	January-97	new	LABCONCO (#20) GCMSSV	N/A
FUME HOOD	MSSV Lab	January-97	January-97	new	LABCONCO (#19)	N/A
COOLER	MSV lab	January-97	January-97	new	TRUE (#22)	1330620
COOLER	MSV lab	January-97	January-97	new	TRUE/GDM-45 (#30)	1-3681854
REFRIG/FREEZER	MSV lab	January-97	January-97	new	KENMORE/2538684012	0983108619
REFRIG/FREEZER	MSV lab	January-97	January-97	new	SEARS (V0A 2)	983108619
FREEZER# 12	MSV lab	January-97	January-97	new	FRIGIDAIRE, MODEL #MFU17F3GW6 (#12)	WB02927861
REFRIGERATOR	MSV lab	January-97	January-97	new	GE (#27)	HR112092
BALANCE	MSV lab	January-97	January-97	new	METTLER AE200	L65273
GCSV 12	GCSV Lab	November-03	November-03	new	AGILENT TECH 6980N	US10338067
GCSV 14	GCSV Lab	January-04	January-04	new	AGILENT TECH 6980N	US10342128
GCSV 15	GCSV Lab	April-04	April-04	new	AGILENT TECH 6980N	CN10413018
GCSV 16	GCSV Lab	August-05	August-05	new	AGILENT TECH 6980N	CN10525006
GCSV 17	GCSV Lab	September-05	September-05	new	AGILENT TECH 6980N	CN10529074

GCAL Equipment List

GCSV 18	GCSV Lab	September-05	September-05	new	AGILENT TECH 6980N	CN10528084
GCSV 19	GCSV Lab	September-05	September-05	new	AGILENT TECH 6980N	CN10534099
GCSV 20	GCSV Lab	October-05	October-05	new	AGILENT TECH 6980N	CN10534109
GCSV 21	GCSV Lab	December-05	December-05	new	AGILENT TECH 6980N	CN10538039
FUME HOOD	GCSV Lab	January-97	January-97	new	LABCONCO(#33)	301116
GCV 5	GCV Lab	February-08	February-08	new	AGILENT 6890 SERIES	US00026701
GCV 6	GCV Lab	November-05	November-05	new	AGILENT 6890N SERIES	CN10538061
GCV 7	GCV Lab	April-07	April-07	new	AGILENT 6890N SERIES	CN10545063
GCV 8	GCV Lab	April-08	April-08	new	AGILENT 6890N SERIES	CN10636089
GCV9	GCV Lab	March-05	June-10	new	AGILENT 6890N	CN10452003
PURGE/TRAP INSTRUMENT 5	GCV Lab	February-08	February-08	new	TELEDYNE TEKMAR / 14-8900-00T	US05257002
PURGE/TRAP INSTRUMENT 6	GCV Lab	November-05	November-05	new	TEKMAR	BETA 005
PURGE/TRAP INSTRUMENT 7	GCV Lab	April-07	April-07	new	TELEDYNE TEKMAR XPT / 14-890000T	US0527002
PURGE/TRAP INSTRUMENT 9	GCV Lab	March-05	June-10	new	TELEDYNE TEKMAR XPT	US0507010
AUTOSAMPLER INSTRUMENT 5	GCV Lab	February-08	February-08	new	TELEDYNE TEKMAR SOLATEK 72	US02277005
AUTOSAMPLER INSTRUMENT 6	GCV Lab	November-05	November-05	new	TELEDYNE TEKMAR AQUATEK 70	US05355004
AUTOSAMPLER INSTRUMENT 7	GCV Lab	April-07	April-07	new	TELEDYNE TEKMAR AQUATEK 70	US05347003
AUTOSAMPLER INSTRUMENT 9	GCV Lab	March-05	June-10	new	TELEDYNE TEKMAR SOLATEK 72	US0324004
Digital Vortex Meter	GCV Lab	November-08	November-08	new	N/A	080801081
BALANCE	GCSV Lab	January-97	January-97	new	SARTORIUS AC211P	50305162
REFRIGERATOR	GCSV Lab	January-97	January-97	new	DANBY #21	N/A
REFRIGERATOR	GCSV Lab	January-97	January-97	new	MASTERBILT (#18)	254034
FREEZER	GCSV Lab	January-97	January-97	new	Frigidaire (#34)	WB44328710
REFRIG/FREEZER	GCSV Lab	January-97	January-97	new	SEARS (#11)	BA01000875
REFRIG/FREEZER	GCSV Lab	January-97	January-97	new	KENMORE (#14)	BA04391055
REFRIG/FREEZER	GCSV Lab	January-97	January-97	new	KENMORE (#15)	BA04391057
REFRIG/FREEZER	GCSV Lab	January-97	January-97	new	KENMORE (#17)	BA03100524
FREEZER	GCSV Lab	January-97	January-97	new	Whirlpool (#37)	EWV3489616
FREEZER	GCSV Lab	January-97	January-97	new	Frigidaire (#26)	WB83724994
HPLC	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
COLUMN HEATER	MSSV Lab	December-06	December-06	new	EPPENDORF TC-45	N/A
HPLC - 2	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - MWD	DE60555127
	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - FLD	DE60555722
	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - ICC	DE60560177
	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - QUANT PUMP	DE60556714
	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - A.I.S	DE60557762
	MSSV Lab	December-06	December-06	new	AGILENT 1200 SERIES - DEGASSER	JP62354304
METALS	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
ICP/MS	Metals Lab	September-10	September-10	new	Agilent 7700/7500 Series	JP10280491
ICP/MS Autosampler	Metals Lab	September-10	September-10	new	Agilent ASX-500	1 S071080A520
ICP	Metals Lab	September-05	September-05	new	PERKIN-ELMER OPTIMA 4300DV	077N0050202
AUTOSAMPLER	Metals Lab	September-05	September-05	new	PERKIN-ELMER AS93 Plus	N.A
ICP	Metals Lab	May-00	May-00	new	PERKIN-ELMER 5300DV	077N5090602
GFAA 2	Metals Lab	January-09	January-09	used	PERKIN-ELMER 800	8111

GCAL Equipment List

GFAA Autosampler	Metals Lab	January-09	January-09	used	PERKIN-ELMER AS800	1852
GFAA Chiller	Metals Lab	January-09	January-09	used	N/A	N/A
HG ANALYZER	Metals Lab	January-97	January-97	new	PERKIN ELMER/FIMS 400	4515
CHILLER	Metals Lab	January-97	January-97	new	Polyscience	G51284
FUME HOOD--FLOW SCIENCES	Metals Lab	January-97	January-97	new	FS3100BK FVA	11-j-07-04
FUME HOOD	Metals Lab	January-97	January-97	new	FS3100BK FVA	11-j-07-15
METALS PREP	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
MICROWAVE	Metals Prep Lab	January-97	January-97	new	CEM MARS5	DS-6208
DIGESTION BLOCKS (4)	Metals Prep Lab	January-97	January-97	new	CPI MOD BLOCK	N/A
FUME HOOD--FLOW SCIENCES (2)	Metals Prep Lab	January-97	January-97	new	FS3100BK DVA / FS3100BK GVA	05-N-03-02 / 08-M-13-02
BALANCE	Metals Prep Lab	July-07	July-07	new	Mettler Toledo XS 104	1128260845
EXTRACTORS	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
FUME HOODS (18)	EXT Area	January-97	January-97	new	N/A	N/A
GLASS WASHER	EXT Area	January-97	January-97	new	AMSCO 400	36911195001
SHAKER (4)	EXT Area	January-97	January-97	new	GLAS-COL 099A	N/A
MILLIPORE(2)	EXT Area	January-97	January-97	new	N/A	N/A
VACUUM PUMP	EXT Area	May-08	May-08	new	Edwards	76434563
GPC (1)	EXT Area	January-97	January-97	new	ABC AP-100	9161SI/AS007-9114-9114
CENTRIFUGE	EXT Area	January-97	January-97	new	IEC HN-SII	N/A
OVEN	EXT Area	January-97	January-97	new	FISHER ISOTEMP 655G	11000184
TCLP/ZHE ROTATOR	EXT Area	January-97	January-97	new	ASSOCIATED DESIGN	NA 05101808
ZHE EXTRACTORS	EXT Area	January-97	January-97	new	ENVIRONMENTAL EXPRESS	NA 05081684
SONICATOR (6)	EXT Area	January-97	January-97	new	FISHER SCIENTIFIC	BBW031-40/BBW0510184/BBW02510184/BBW05101850
INCUBATOR SHAKER	EXT Area	January-97	January-97	new	NEW BRUNSWICK SCIENTIFIC/CLASSIC SEI	100524881
BALANCE	EXT Area	January-97	January-97	new	METTLER PM 3000	M33557
BALANCE	EXT Area	January-97	January-97	new	METTLER PG 3001 S	1117331005
BALANCE	EXT Area	January-97	January-97	new	AND FX-300	5015502
BALANCE	EXT Area	January-97	January-97	new	OHAUS SCOUT PRO SP2001	7124330243
BALANCE	EXT Area	January-97	January-97	new	OHAUS SCOUT PRO SP402	7124280031
BALANCE	EXT Area	January-97	January-97	new	OHAUS SCOUT PRO SPE4001	7123450167
BALANCE	EXT Area	January-97	January-97	new	OHAUS SCOUT PRO SP2001	7124371673
PH METER	EXT Area	January-97	January-97	new	ORION SA520	QT20A
PH METER	EXT Area	January-97	January-97	new	THERMO ORION 720A	074216
PH METER	EXT Area	January-97	January-97	new	ORION 720A+	085153
PH METER	EXT Area	January-97	January-97	new	ORION 720A+	089622
PH PROBE 01-A (C PROBE	EXT Area	January-97	January-97	new	ORION SURE-FLOW ROSS 8172BNWP	LYI-16730
PH PROBE 03-A (C PROBE	EXT Area	January-97	January-97	new	ORION SURE-FLOW ROSS 8172BNWP	MX3-10451
PH ELECTRODE (03)	EXT Area	January-97	January-97	new	ORION SURE FLOW	MP3-10011
COOLER	EXT Area	January-97	January-97	new	TRUE (#5)	1334961
REFRIG/FREI / I R	EXT Area	January-97	January-97	new	WHITE WESTINGHOUSE (#8)	LAI0903763
REFRIG/FREI / I R	EXT Area	January-97	January-97	new	WHITE WESTINGHOUSE (#9)	BA04391056
REFRIG/FREI / I R	EXT Area	January-97	January-97	new	Frigidaire (R#34)	BA61019273
FREEZER	EXT Area	January-97	January-97	new	WHITE WESTINGHOUSE (#23)	WB40802629
ULTRASONIC CLEANER	EXT Area	January-97	January-97	new	FISHER SCIENTIFIC/FS30	RTB040265340

GCAL Equipment List

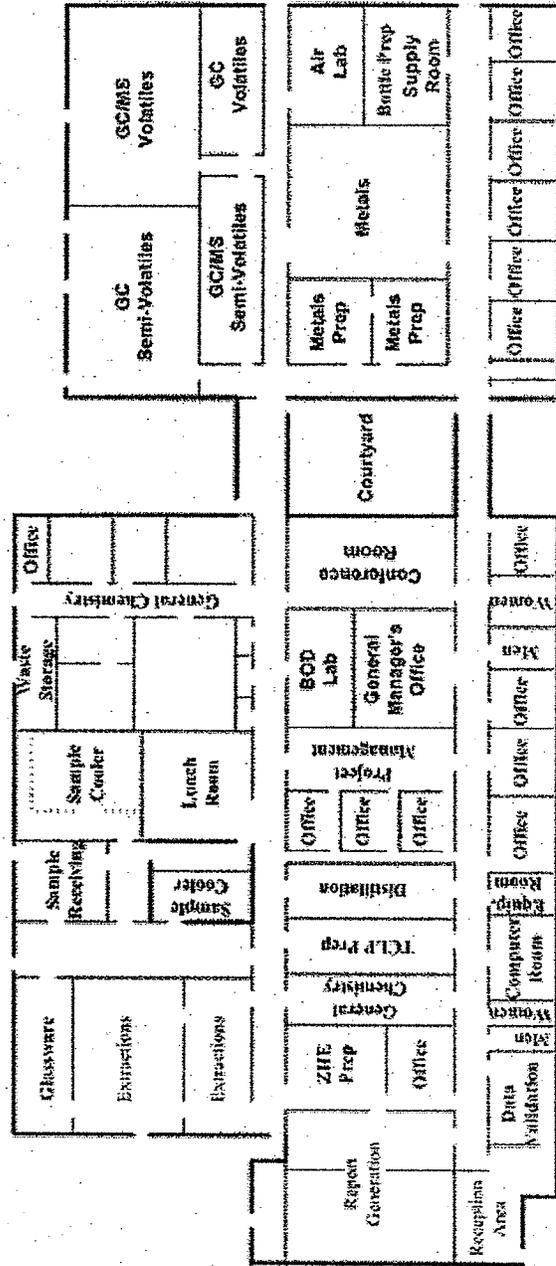
	EXT Area	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
MUFFLE FURNACE	EXT Area	January-97	January-97	new	FISHER SCIENTIFIC/ISOTEMP 550 SERIES M	410N0074
STANDARD TEST SIEVE	EXT Area	January-97	January-97	new	WS TYLER/MODEL #RX-812	24372
PYROMETER	EXT Area	January-09	January-09	new	PM20700	SN 8502832
SAMPLE CONCENTRATOR	EXT Area	January-05	January-05	new	OA-SYS	SN 20018
SAMPLE CONCENTRATOR	EXT Area	January-05	January-05	new	OA-SYS	SN 20019
WET CHEMISTRY	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER
TOX	Wet Chemistry	January-97	January-97	new	MITSUBISHI TOX-10E	N/A
TOC (1)	Wet Chemistry	June-08	June-08	new	Shimadzu TOC-V CSH	H51104535288CS
	Wet Chemistry	January-97	January-97	new	Shimadzu Solid Module	H52504500370
	Wet Chemistry	January-97	January-97	new	Shimadzu Autosampler	H52104502483
AUTOANALYZER	Wet Chemistry	June-08	June-08	new	LACHET QUICK CHEM AE	200-474
IC	Wet Chemistry	October-00	October-00	new	DIONEX LC20/ED40/AD20/AS40	900915
COD REACTOR	Wet Chemistry	January-97	January-97	new	HACH COD REACTOR	910404575/920800007697
TURBIDIMETER	Wet Chemistry	January-97	January-97	new	HACH 2100P	960700011424
SPECTROPHOTOMETER	Wet Chemistry	January-97	January-97	new	HACH #3 DR 2800	1209697
SPECTROPHOTOMETER	Wet Chemistry	January-97	January-97	new	HACH #4 DR 2800	11996-79
TITRATOR	Wet Chemistry	January-97	January-97	new	METTLER TOLEDO DL53	S119484414
VISCOMETER	Wet Chemistry	January-97	January-97	new	BROOKFIELD DVII	32587
WATER BATH	Wet Chemistry	January-97	January-97	new	BROOKFIELD TC-200	GCAL# 0311
CLOSED CUP FLASH PT	Wet Chemistry	January-97	January-97	new	PRECISION SCIENTIFIC	10BR-12
FLASHPOINT (FP3)	Wet Chemistry	December-08	December-08	new	Herzog HFP 339	083390442
AMMONIA PROBE	Wet Chemistry	January-97	January-97	new	ORION 95-12	N/A
OXYGEN BOMB CAL	Wet Chemistry	January-97	January-97	new	PARR	6616
CONDUCTIVITY METER	Wet Chemistry	September-02	September-02	new	OAKTON pH/CON 510 SERIES	79134
COLIFORM BATH 1	Wet Chemistry	January-92	January-92	new	PRECISION SCIENTIFIC INCUBATOR	10AZ-1
COLIFORM BATH 2	Wet Chemistry	May-08	May-08	new	THERMOSCIENTIFIC INCUBATOR	204785
COLIFORM BATH 3	Wet Chemistry	May-09	May-09	new	THERMOSCIENTIFIC INCUBATOR	207063
PH METER	Wet Chemistry	January-97	January-97	new	ORION 720A+	092891
PH PROBE 04/ATC PROBE	Wet Chemistry	January-97	January-97	new	ORION SURE-FLOW ROSS 8102BNUWP	LUI-18038
PH METER	Wet Chemistry	January-97	January-97	new	ORION 420A	7881
PH PROBE	Wet Chemistry	January-97	January-97	new	ORION TRIODE	N/A
PH PROBE/ATC PROBE	Wet Chemistry	January-97	January-97	new	ORION COMB 915600/917006	N/A
PH METER (PH-05)	Wet Chemistry	October-08	October-08	new	ORION 2STAR	B11765
PH PROBE	Wet Chemistry	January-97	January-97	new	ORION 9107APMD	RMR21
PH Meter (PH-07)	Wet Chemistry	September-10	September-10	new	Thermo Scientific/Dual Star	E03025
AUTOCCLAVE	Wet Chemistry	September-10	September-10	new	Tuttnauer	I0003285
DO METER	Wet Chemistry	January-97	January-97	new	YSI MODEL 59/5903 PROBE	93A01946
DO METER	Wet Chemistry	March-10	March-10	new	YSI MODEL 5100	10A 101264
OVENS (2)	Wet Chemistry	January-97	January-97	new	BLUE M SW17TA-1	SW-5478/SW-5408
OVEN	Wet Chemistry	January-97	January-97	new	FISHER SCIENTIFIC 3510-ISF	1879070606147
OVEN	Wet Chemistry	January-97	January-97	new	GRIEVE PL-326	444341
OVEN	Wet Chemistry	January-97	January-97	new	FISHER SCIENTIFIC ISOTEMP/MODEL 516G	506N0196
OVEN	Wet Chemistry	January-97	January-97	new	FISHER SCIENTIFIC ISOTEMP/MODEL 650G	508N0138
OVEN	Wet Chemistry	January-97	January-97	new	FISHER SCIENTIFIC ISOTEMP/MODEL 650G	508N0137

GCAL Equipment List

FURNACE	EXT area	January-97	January-97	January-97	new	THERMOLYNE 1500	N/A
DESSICATORS (7)	Wet Chemistry	January-97	January-97	January-97	new	DRY KEEPER	N/A
INCUBATOR	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC(#3)	WB93928030
BALANCE	Wet Chemistry	January-97	January-97	January-97	new	METTLER AE160	C05693
BALANCE	Wet Chemistry	January-97	January-97	January-97	new	METTLER AX504	1122043050
BALANCE	Wet Chemistry	January-97	January-97	January-97	new	SARTORIUS / PT6-000V2	60802675
BALANCE	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC / ACCU-224	F224075004
FUME HOODS (5)	Wet Chemistry	January-97	January-97	January-97	new	N/A	N/A
INCUBATOR (BOD#6)	Wet Chemistry	January-97	January-97	January-97	new	REVCO	T28C-142152-TC
INCUBATOR (BOD#2)	Wet Chemistry	January-97	January-97	January-97	new	PRECISION SCIENTIFIC	10AZ-12
INCUBATOR (BOD#7)	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC	407N0211
INCUBATOR (BOD#5)	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC	WB93928030
INCUBATOR (BOD#8)	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC	WB53337398
INCUBATOR (BOD#9)	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC	2018080398339
INCUBATOR (BOD#10)	Wet Chemistry	January-97	January-97	January-97	new	FISHER SCIENTIFIC	2018080607337
REFRIG/FREEZER	Wet Chemistry	January-97	January-97	January-97	new	SEARS (#19)	LA91905856
REFRIG/FREEZER	Wet Chemistry	January-97	January-97	January-97	new	WHITE WESTINGHOUSE (#1)	WB10507478
VACUUM PUMP	EXT Area	May-08	May-08	May-08	new	Edwards	7643558
GRINDING MILL	Wet Chemistry	January-97	January-97	January-97	new	THE STRAUB CO./MODEL 4E	N/A
LOG-IN	Location	Date Received	Date in Service	Condition	MAKE/MODEL	SERIAL NUMBER	
FUME HOOD (1)	LOGIN Area	January-97	January-97	new	FUME HOOD #9	N/A	
COOLER	LOGIN Area	January-97	January-97	new	TRUE (#2)	708391	
COOLER	LOGIN Area	January-97	January-97	new	TRUE (#3)/GDM-72	1-3792963	
WALK-IN COOLER (2)	LOGIN Area	January-97	January-97	new	N/A	N/A	
REFRIG/FREEZER	LOGIN Area	January-97	January-97	new	KENMORE (#4)	BA01902878	
IR THERMOMETER	LOGIN Area	May-08	May-08	new	FISHER SCIENTIFIC	72704761	
REFRIG/FREEZER	LOGIN Area	January-97	January-97	new	FRIGIDAIRE (#33)	BA454622395	
FREEZER	LOGIN Area	January-97	January-97	new	FRIGIDAIRE(#27) MFU17F3GW6	WB03102969	
SCANNER	LOGIN Area	January-97	January-97	new	HEWLETT PACKARD SCANJET 5470C/C9850	CN1B41HOTZ	
FREEZER	LOGIN Area	January-97	January-97	new	FRIGIDAIRE (#28) FFU20FC4CWO	WB34937210	
FREEZER	LOGIN Area	January-97	January-97	new	FRIGIDAIRE (#29) FFC15C4CWO	WB40427812	

Appendix D

Facility Floor Plan



Approximate Square Footage: 19,600 Sq. Ft.

Scale: 1 in. = 25 ft.

Sample Receiving/Cooler:	1320	GC/MS Volatiles:	906
Extractions/Prep:	2913	GC/MS Semi-Volatiles:	394
General Chemistry:	3025	GC Volatiles:	323
Metals:	840	GC Semi-Volatiles:	906
Metals Preparation:	450	Report Generation:	713
Supply Room:	616	Offices/Storage:	8200

Appendix E

GULF COAST ANALYTICAL LABORATORIES CERTIFICATIONS

Scopes of Accreditation are maintained by the QA/QC department and are available for review upon request.

- < State of Illinois Environmental Protection Agency, NELAP Accreditation #200048, Certification No. 001202 (Expiration: February 2011)
- < State of California Environmental Laboratory Accreditation Program, (Expiration: May 2011)
- < State of Florida, Department of Health, Bureau of Laboratories, NELAP, Lab ID: E87854 (Expiration: June 2011)
- < South Carolina Department of Health and Environmental Control, Environmental Laboratory Certification Program, Certificate Number: 73006001 (Expiration: June 2011)
- < Louisiana Department of Environmental Quality, Environmental Laboratory Accreditation Program, NELAP, Certificate Number 01955 (Expiration: June 2011)
- < Texas Commission on Environmental Quality, Certificate Number: T104704178-05-TX (Expiration: August 2011)
- < State of Arkansas, Department of Pollution Control and Ecology, Laboratory Certification Program (Expiration: August 2011)
- < Oklahoma Department of Environmental Quality, Laboratory Certification Program, ID # 9403 (Expiration: August 2011)
- < State of Kansas, Department of Health and Environment, NELAP, Certificate No. E-10354 (Expiration: October 2011)
- < Arizona Department of Health Services, License AZ0718 (Expiration November 2011)
- < State of North Carolina, Division of Water Quality Laboratory Certification Program, Certificate 618 (December 2010)
- < DoD ELAP, Certificate ADE-1482 (Expires September 2012)
- < State of Georgia Environmental Protection Division accreditation based on LA NELAP

Appendix F

GULF COAST ANALYTICAL LABORATORIES, INC
STANDARD OPERATING PROCEDURES

<u>SOP</u>	<u>METHOD</u>	<u>REVISION #</u>	<u>DATE</u>	<u>LAST REVIEW</u>
<u>Extractions</u>				
EXT-001	BNA Solid/Low/3550	16	06/14/2010	
EXT-002	Pest/PCB Low/3550	16	12/23/2009	
EXT-003	BNA Prep SEP Funnel/3510	19	07/08/2010	
EXT-004	BNA Prep Continuous/3520	8	01/04/2010	
EXT-010	Pest/PCB Prep SEP Fun/3510	14	04/07/2010	
EXT-011	Pest/PCB Prep Cont/3520	8	01/04/2010	
EXT-017	Herbicide Preparation	20	12/21/2009	
EXT-019	ZHE Cleanup	6	01/06/2010	
EXT-026	TCLP/1311	8	01/06/2010	
EXT-027	TPHD Solid/Low Level Ext	13	09/13/2010	
EXT-029	TPHD Prep SEP Funnel	11	09/13/2010	
EXT-031	Herbicide Prep Soils	16	08/16/2010	
EXT-032	pH-Solids & Wastes	9	12/18/2009	
EXT-033	pH-Waters	8	12/18/2009	
EXT-034	Orgphos Pest Soil Prep/8141	11	12/22/2010	
EXT-035	Orgphos Pest/SEP Fun/8141	10	12/22/2010	
EXT-036	TCLP-Volatiles/1311	7	01/06/2010	
EXT-037	Pest-Organic Prep	8	12/22/2009	
EXT-038	BNA-Organic Prep	7	12/22/2009	
EXT-050	BNA Sep OLC0.2	6	01/05/2010	
EXT-051	Pest/PCB OLC0.2	5	01/05/2010	
EXT-052	Oil & Grease-1664	14	08/26/2010	
EXT-056	PAH/Soil/3550	7	12/23/2009	
EXT-057	PAH/Water/3510	7	12/18/2009	
EXT-058	Sulfuric Acid/Permanganate Clean-up	4	01/05/2010	
EXT-059	Florisil Clean-up	4	01/05/2010	
EXT-064	Oil & Grease/Soxhlet-29B	7	08/26/2010	
EXT-065	Explosives Prep/Soil Samples	8	01/04/2010	
EXT-066	Explosives Prep/Water Samples	7	04/22/2010	
EXT-068	Formaldehyde Derivatization/Soil	4	01/04/2010	
EXT-069	Formaldehyde Derivatization/Water	5	12/22/2009	
EXT-070	SPLP/1312 Water & Solids	3	02/01/2010	
EXT-071	SPLP-Volatiles/1312 Water & Solids	1	10/09/2003	
EXT-073	Extraction of Tissue Samples	3	07/14/2009	
EXT-074	% Lipids in Tissue	4	08/25/2010	
EXT-076	625 SIM/Water	4	12/21/2009	
EXT-077	Method 619 Extraction	4	08/16/2010	
EXT-078	Formaldehyde Extraction	1	12/22/2009	
<u>GC</u>				
GC-002	Volatiles/602	8	11/18/2009	
GC-004	DRO/ORO	15	05/24/2010	
GC-006	Gasoline Range Organics	16	09/13/2010	

GULF COAST ANALYTICAL LABORATORIES, INC
STANDARD OPERATING PROCEDURES

<u>SOP</u>	<u>METHOD</u>	<u>REVISION #</u>	<u>DATE</u>	<u>LAST REVIEW</u>
GC-007	TPH by Texas 1005/1006	8	09/10/2010	
GC-008	Organophosphorous Pest/8141	7	05/24/2010	
GC-011	Chlorinated Herbicides/8151	9	05/21/2010	
GC-012	Pesticides/PCB 608	7	06/23/2009	
GC-013	Pesticides/8081B	13	05/24/2010	
GC-022	BTEX/8021	7	05/21/2010	
GC-023	PCB/8082	11	05/24/2010	
GC-024	Dissolved Gas/RSK-175	6	05/21/2010	
GC-025	EPH-Extractable Pet. Hydrocarbons	5	09/10/2009	
GC-028	Methanol/NCASI Method	2	09/17/2009	
GC-029	Methanol/Method 8015B	6	07/27/2010	
GC-031	Florida PRO	6	05/24/2010	
GC-032	Volatile Petroleum Hydrocarbons	2	07/10/2009	
GC-033	Triazine Pesticides – Method 619	2	12/01/2009	
GC-034	Method 8011	6	05/24/2010	
 <u>GCMSSV</u>				
GCMSSV-001	Semivolatiles/8270C	16	05/21/2010	
GCMSSV-002	Semivolatiles/625	11	11/10/2010	
GCMSSV-003	Semivolatiles/625 SIM	3	05/21/2010	
GCMSSV-004	Semivolatiles/8270D	4	09/13/2010	
GCMSSV-005	Parent/Alkyl PAH	0	09/27/2010	
 <u>GCMSV</u>				
GCMSV-002	Volatiles/624	9	05/21/2010	
GCMSV-003	Volatiles/8260	20	07/09/2010	
GCMSV-004	Oxygenates/8260	0	10/07/2008	
 <u>General Laboratory</u>				
GEN-001	Laboratory Glassware Prep.	5	10/02/2006	1/28/2010
GEN-002	Balance Calibration	8	05/05/2010	
GEN-003	Temperature Monitoring	10	04/30/2010	
GEN-005	Mechanical Pipette Calibration	5	09/29/2006	1/28/2010
GEN-006	Standard Preparation	7	06/17/2010	
GEN-007	GCAL Training	6	08/26/2010	
GEN-008	Documentation of Data (Logbooks)	6	06/26/2002	1/28/2010
GEN-009	Waste Handling	7	07/13/2010	
GEN-010	General Lab Monitoring	7	05/11/2010	
GEN-012	Preventive Maintenance	4	02/02/2010	
GEN-013	Spill Clean Up	1	07/23/1998	1/28/2010
GEN-015	Laboratory Contingencies	1	08/31/2000	1/27/2010
GEN-016	Definitions	2	05/09/2005	
GEN-018	Non-conformances/Corrective Actions	5	05/14/2010	
GEN-019	Project Specific Requirements	2	03/10/2010	

**GULF COAST ANALYTICAL LABORATORIES, INC
STANDARD OPERATING PROCEDURES**

<u>SOP</u>	<u>METHOD</u>	<u>REVISION #</u>	<u>DATE</u>	<u>LAST REVIEW</u>
GEN-020	Standard Tracking	1	05/06/2010	
GEN-021	Calibration Modules for Target	0	05/26/2009	
GEN-022	Linear Least Square Regression	0	05/26/2009	
<u>HPLC</u>				
HPLC-001	PAH'S/8310	10	05/21/2010	
HPLC-003	Explosives/8330	7	05/25/2010	
HPLC-004	Carbonyls by HPLC (8315A)	4	04/01/2009	
<u>Lab Administration/General</u>				
LAD-001	Master Signature List	3	07/14/1998	1/26/2010
LAD-002	Visitor Procedure	4	02/26/2003	11/09/2009
LAD-003	Report Generation	8	07/19/2010	
LAD-005	Vehicle Inspection	3	07/14/1998	11/16/2009
LAD-008	Postage Machine	7	11/10/2009	
LAD-009	Mail	7	11/10/2009	
LAD-011	Answering the Telephone	7	11/09/2009	
LAD-012	Sending a Fax	6	09/15/2006	11/09/2009
LAD-013	Receptionist/Arriving and Departing	9	11/10/2009	
LAD-014	Project Management	9	06/08/2010	
LAD-018	Confidentially	4	03/15/2007	11/13/2009
LAD-019	Data Assembly	0	09/13/2006	
<u>Metals</u>				
MET-001	Glassware Prep-Metals	8	11/09/2009	
MET-002	Digestion for GFAAS	10	10/19/2009	
MET-003	Digestion for As & Se-GFAAS	6	10/19/2009	
MET-004	Digestion Solids/3050	13	11/09/2009	
MET-005	Digestion for ICP-water	13	11/09/2009	
MET-006	Digestion for Mercury	19	05/19/2010	
MET-008	Mercury Analysis/PS200	16	05/19/2010	
MET-010	ICP Analysis/PE 3000XL/4300DV	18	05/19/2010	
MET-013	Hardness/Calc. Method	5	11/02/2009	
MET-015	GFAAS Analysis/PE 4100ZL	6	05/19/2010	
MET-018	Microwave Digestion/Organics	4	11/09/2009	

GULF COAST ANALYTICAL LABORATORIES, INC
STANDARD OPERATING PROCEDURES

<u>SOP</u>	<u>METHOD</u>	<u>REVISION #</u>	<u>DATE</u>	<u>LAST REVIEW</u>
MET-019	Total Recoverable Metals	2	11/09/2009	
MET-020	ICP Water Preparation for 200.7	2	11/02/2009	
<u>Quality Assurance</u>				
QA-001	Document Control	9	08/10/2010	
QA-002	Data Reduction Validation	7	07/08/2010	
QA-003	Report Validation	5	7/19/2010	
QA-004	Laboratory Audits	3	05/09/2005	1/29/2010
QA-007	Data Archive	5	06/14/2010	
QA-008	Generation of SOP's	4	01/31/2007	1/27/2010
QA-009	Determination of MDL's	9	01/30/2007	1/26/2010
QA-010	Proper Handling of Raw Data	4	11/02/2006	1/26/2010
QA-011	Data Integrity and Ethical Practices	3	08/28/2004	1/25/2010
QA-012	Control Charts	3	07/12/2010	
QA-013	Estimation of Uncertainty	2	06/14/2010	
QA-014	Demonstration of Capability	2	06/10/2010	
QA-015	Performance Evaluation Studies	1	06/17/2010	
<u>Sample Administration</u>				
SAD-001	Sample Log-In	15	08/02/2010	
SAD-002	Sample Custodian	9	08/12/2008	11/16/2009
SAD-003	Sample Kit Preparation	8	08/14/2008	11/16/2009
SAD-004	LIMS Log-In Procedure	8	05/31/2006	11/16/2009
SAD-006	Sample Couriers	0	11/17/2010	
<u>Wet Lab (General Chemistry)</u>				
WL-002	Total Solids	9	11/09/2009	
WL-003	Total Dissolved Solids	9	11/05/2009	
WL-004	Total Suspended Solids	11	11/05/2009	
WL-005	Vol. Suspended Solids	7	11/05/2009	
WL-006	Sulfate/Turbidimetric	9	05/18/2010	
WL-007	Sulfite	6	10/29/2009	
WL-008	Color	7	05/18/2010	
WL-009	Paint Filter Test	9	11/09/2009	
WL-012	Hexavalent Chromium	8	05/18/2010	
WL-014	Phenols	9	05/18/2010	
WL-015	Cyanide	9	05/18/2010	
WL-016	Chlorine	7	05/18/2010	
WL-017	Fluoride	7	05/18/2010	
WL-018	BOD	20	05/19/2010	
WL-019	Specific Gravity	8	10/29/2009	

GULF COAST ANALYTICAL LABORATORIES, INC
STANDARD OPERATING PROCEDURES

<u>SOP</u>	<u>METHOD</u>	<u>REVISION #</u>	<u>DATE</u>	<u>LAST REVIEW</u>
WL-021	COD/HACH	10	05/18/2010	
WL-022	Ash/D482-80	9	11/09/2009	
WL-025	Surfactant	7	05/18/2010	
WL-026	Fecal Coliform	11	07/20/2009	
WL-028	Silica	7	05/18/2009	
WL-029	Specific Conductance	7	02/17/2010	
WL-032	Turbidity	9	11/04/2010	
WL-033	Sulfide/MB	9	06/07/2010	
WL-034	Total Phosphorus	8	05/18/2010	
WL-035	Total Settable Solids	4	10/29/2009	
WL-037	TOX	4	11/03/2009	
WL-038	Chlorides	8	05/18/2010	
WL-041	Nitrate/Nitrite/N+N	9	05/18/2010	
WL-042	Anions by Ion Chromatography	15	05/19/2010	
WL-043	TOC Waters	10	08/26/2010	
WL-044	Heat of Combustion	7	11/02/2009	
WL-045	TKN/Titration	8	09/13/2010	
WL-046	NH3 by ISE	10	11/09/2009	
WL-047	Ferrous Iron	5	05/18/2010	
WL-048	Orthophosphate	9	05/18/2010	
WL-051	Sulfide Titration	7	11/17/2009	
WL-052	Viscosity	3	06/15/2009	
WL-054	Reactive Cyanide & Sulfide	9	05/18/2010	
WL-056	Corrosivity Towards Steel	6	12/17/2009	
WL-057	TOC Solid	3	10/11/2010	
WL-060	Flashpoint-Automated	5	10/28/2009	
WL-061	Water by Karl Fisher	2	10/29/2009	
WL-062	Automated Ammonia Titration	6	09/13/2010	
WL-063	Automated Alkalinity	6	11/09/2009	
WL-064	Automated Low Alkalinities	5	11/10/2009	
WL-066	Ignitability of Solids	3	02/18/2009	11/05/2009
WL-068	Perchlorate	2	01/11/2010	
WL-069	Total Acidity	2	10/29/2009	
WL-070	Volatile Fatty Acids	1	11/04/2009	
WL-073	Desiccator Monitoring	1	11/09/2009	

Appendix G

QAPP for Tissue Homogenization, Preparation and Analysis

Introduction

GCAL has performed tissue analysis for over a decade in support of remedial investigation and risk assessment studies. The analytical analysis of tissues is performed over a wide range of tests, including trace metals and mercury, PAHs, PCBs, and lipid determination. GCAL is accredited through the Louisiana DEQ for tissue analysis with an extensive scope of accreditation in this area.

Each type of tissue sample represents a unique challenge due to the sample matrix. GCAL has developed methods to deal with tissue homogenization, sample prep, clean-up, and analysis of tissue samples. All lab procedures are documented in laboratory SOPs. The analytical techniques follow SW-846 methodologies; including instrument calibration, QC requirements, and detection limit studies. All analysts performing sample prep and analysis must complete a demonstration of capability.

Sample Receipt and Chain of Custody

Samples are received at the laboratory, generally on ice, and following all sample receipt requirements outlined in SOP SAD-001. All samples are logged into the LIMS system, and given unique sample identification. Samples will then be transferred to the laboratory for homogenization or stored in a temperature controlled and monitored freezer. GCAL uses a bar code system for sample chain of custody and tracking, which is stored in the LIMS from receipt through disposal. Unused samples are stored frozen for a period of 60 days following receipt unless other arrangements are agreed upon. Please note that GCAL does not accept samples that require radiation treatment to destroy potential biological hazards such as rabies.

Tissue Homogenization

All tissue samples are subjected to homogenization and/or dissection techniques prior to analysis, which are designed to ensure a representative sub-sample is obtained for each analytical parameter. The procedures used may vary significantly depending on the tissue type, the technical specifications of the project, and the requested analysis. Special blades are employed when metals analysis is requested to avoid contamination of samples. Following homogenization, a representative sub-sample is placed in a labeled pre-cleaned jar(s) and transferred to the appropriate departments for sample prep and analysis. Any unused portions are labeled and frozen.

Tissue Prep for Semi-volatile Organic Analysis

Sample prep is dependant on the project requirements including detection limits. The sample weight and final extract volume is carefully selected in order to achieve requested reporting limits and limit sample matrix interferences. Homogenized tissues are thawed, and then further homogenized by grinding in a mortar and pestle and mixing with C18. Samples are then extracted with solvent. Clean-up procedures selected depend on the requested analysis and sample matrix. These may include silica gel clean-up for PAHs and acid clean-up for PCBs. All samples are prepped with quality control samples, including method blanks, blank spikes (LCS), and matrix spikes (if sample volume is sufficient). All samples are spiked with surrogate standards as required by the method.

Lipids Determination

Lipids are analyzed gravimetrically. A homogenized sample is weighed and extracted in methylene chloride using sonication. The extract is allowed to air dry and the residue is weighed. Results are reported as percent lipids.

Tissue Prep for Trace Metal and Mercury Analysis

Tissue prep of trace metals is typically performed in a closed Teflon vessel under high temperature and pressure. For mercury, the laboratory follows similar procedures as for other solids, except the initial sample weight is larger (typically 6 grams). This allows a more representative sub-sample for tissues. The digestion procedure is carried out using similar ratios of digesting/oxidizing reagents as suggested in SW-846 procedures to account for the larger sample volume.

Sample Analysis

There are typically no modifications needed for the analysis of tissue extracts/digestates. Procedures are outlined in laboratory SOPs.

PRESERVATION CHECKLIST / COOLER RECEIPT

Gulf Coast Analytical Laboratories, Inc.

WO: 210111016

Type: M

Desc:

Report: REVIEW_RPT

Work ID: SWMU54 - 150+ Post Inj 2nd Qtr

Status: WP

Project Seq: 110959

Created: 11/10/2010 10:58

Client: 4380 - CH2M Hill Constructors

QA:

Profile: 152015 - PR-SWMU54/55 - Puerto Rico-SWMU54/55

PO: 812168

WORKORDER SAMPLES

pH PRESERVATIVE

VOA HEADSPACE

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101601-1	40	HCL							OK
21011101601-10	40	HCL							OK
21011101601-11	40	HCL							OK
21011101601-2	40	HCL							OK
21011101601-3	40	HCL							OK
21011101601-4	40	NONE			X				OK
21011101601-5	40	NONE			X				OK
21011101601-6	40	NONE			X				OK
21011101601-7	OC	NONE			X			X	OK
21011101601-8	OC	NONE			X			X	OK
21011101601-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101602-1	40	HCL							OK
21011101602-10	40	HCL							OK
21011101602-11	40	HCL							OK
21011101602-2	40	HCL							OK
21011101602-3	40	HCL							OK
21011101602-4	40	NONE			X				OK
21011101602-5	40	NONE			X				OK
21011101602-6	40	NONE			X				OK
21011101602-7	OC	NONE			X			X	OK
21011101602-8	OC	NONE			X			X	OK
21011101602-9	OC	Zn Ac						X	OK

pH PRESERVATIVE VOA HEADSPACE

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101603-1	40	HCL							OK
21011101603-10	40	HCL							OK
21011101603-11	40	HCL							OK
21011101603-2	40	HCL							OK
21011101603-3	40	HCL							OK
21011101603-4	40	NONE			X				OK
21011101603-5	40	NONE			X				OK
21011101603-6	40	NONE			X				OK
21011101603-7	OC	NONE			X			X	OK
21011101603-8	OC	NONE			X			X	OK
21011101603-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101604-1	40	HCL							OK
21011101604-10	40	HCL							OK
21011101604-11	40	HCL							OK
21011101604-2	40	HCL							OK
21011101604-3	40	HCL							OK
21011101604-4	40	NONE			X				OK
21011101604-5	40	NONE			X				OK
21011101604-6	40	NONE			X				OK
21011101604-7	OC	NONE			X			X	OK
21011101604-8	OC	NONE			X			X	OK
21011101604-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101605-1	40	HCL							OK
21011101605-10	40	HCL							OK
21011101605-11	40	HCL							OK
21011101605-2	40	HCL							OK
21011101605-3	40	HCL							OK
21011101605-4	40	NONE			X				OK
21011101605-5	40	NONE			X				OK
21011101605-6	40	NONE			X				OK
21011101605-7	OC	NONE			X			X	OK
21011101605-8	OC	NONE			X			X	OK
21011101605-9	OC	Zn Ac						X	OK

pH PRESERVATIVE

VOA HEADSPACE

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101606-1	40	HCL							OK
21011101606-10	40	HCL							OK
21011101606-11	40	HCL							OK
21011101606-2	40	HCL							OK
21011101606-3	40	HCL							OK
21011101606-4	40	NONE			X				OK
21011101606-5	40	NONE			X				OK
21011101606-6	40	NONE			X				OK
21011101606-7	OC	NONE			X			X	OK
21011101606-8	OC	NONE			X			X	OK
21011101606-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101607-1	40	HCL							OK
21011101607-10	40	HCL							OK
21011101607-11	40	HCL							OK
21011101607-2	40	HCL							OK
21011101607-3	40	HCL							OK
21011101607-4	40	NONE			X				OK
21011101607-5	40	NONE			X				OK
21011101607-6	40	NONE			X				OK
21011101607-7	OC	NONE			X			X	OK
21011101607-8	OC	NONE			X			X	OK
21011101607-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101608-1	40	HCL							OK
21011101608-10	40A	HCL						X	OK
21011101608-11	40A	HCL						X	OK
21011101608-2	40	HCL							OK
21011101608-3	40	HCL							OK
21011101608-4	40	NONE			X				OK
21011101608-5	40	NONE			X				OK
21011101608-6	40	NONE			X				OK
21011101608-7	OC	NONE			X			X	OK
21011101608-8	OC	NONE			X			X	OK
21011101608-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101609-1	40	HCL							OK
21011101609-10	40A	HCL						X	OK
21011101609-11	40A	HCL						X	OK
21011101609-2	40	HCL							OK
21011101609-3	40	HCL							OK
21011101609-4	40	NONE			X				OK
21011101609-5	40	NONE			X				OK
21011101609-6	40	NONE			X				OK
21011101609-7	OC	NONE			X			X	OK
21011101609-8	OC	NONE			X			X	OK
21011101609-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101610-1	40	HCL							OK
21011101610-10	40A	HCL						X	OK
21011101610-11	40A	HCL						X	OK
21011101610-2	40	HCL							OK
21011101610-3	40	HCL							OK
21011101610-4	40	NONE			X				OK
21011101610-5	40	NONE			X				OK
21011101610-6	40	NONE			X				OK
21011101610-7	OC	NONE			X			X	OK
21011101610-8	OC	NONE			X			X	OK
21011101610-9	OC	Zn Ac						X	OK

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101611-1	40	HCL							OK
21011101611-10	40A	HCL						X	OK
21011101611-11	40A	HCL						X	OK
21011101611-2	40	HCL							OK
21011101611-3	40	HCL							OK
21011101611-4	40	NONE			X				OK
21011101611-5	40	NONE			X				OK
21011101611-6	40	NONE			X				OK
21011101611-7	OC	NONE			X			X	OK
21011101611-8	OC	NONE			X			X	OK
21011101611-9	OC	Zn Ac						X	OK

pH PRESERVATIVE

VOA HEADSPACE

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101612-1	40	HCL	<input type="checkbox"/>	OK					
21011101612-2	40	HCL	<input type="checkbox"/>	OK					
21011101612-3	40	HCL	<input type="checkbox"/>	OK					

Container ID	Type	Preservative	pH PRESERVATIVE			VOA HEADSPACE			CONTAINER CONDITION
			A	U	N/A	A	U	N/A	
21011101613-1	40	HCL	<input type="checkbox"/>	OK					
21011101613-2	40	HCL	<input type="checkbox"/>	OK					
21011101613-3	40	HCL	<input type="checkbox"/>	OK					

A = ACCEPTABLE

U = UNACCEPTABLE

N/A = NOT APPLICABLE

COOLER (S) TEMPERATURE

A

U

LIMIT = 4C + \ - 2C

MAXIMUM VOLATILE HEADSPACE BUBBLE 6MM

Custody Seal

used Yes No

in tact Yes No

LABEL(S)

VERIFIED _____

CUSTODIAN _____

SYSTEM & TECHNICAL AUDIT

PARAMETER:

DATE:

METHODS:

AUDITOR:

ANALYST(S)

INTERVIEWED:

SOP'S:

Are the SOP's readily available, and do the analysts know where they are?

Are the personnel familiar with the contents of the SOP?

Does the SOP describe the procedures actually being employed within the laboratory?

Has the SOP been reviewed this year?

OTHER:

Are the MDL studies less than one year old?

Are all analysts adequately trained and certified to perform the tests?

DISCREPANCES & COMMENTS:

QA/QC representative

Internal Audit-Training Files

Analyst: _____
Year Reviewed: _____
Section: _____

Is initial lab and safety training complete?

Yes No

Is ethics training up-to-date as shown by a signed data integrity agreement? (Initial training and annual)

Yes No

Is manual integration training up-to-date as shown by a signed manual integration policy? (Annual training for all organic sections and analysts performing IC)

Yes No

Are there signed DOC on file for each test and matrix performed?

Yes No

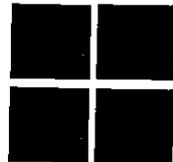
Is there a Read and Understand Form completed for each corresponding SOP and the QAPP?

Yes No

Describe deficiencies found:

Reviewed by: _____

Date: _____

GCAL 

GULF COAST ANALYTICAL LABORATORIES, INC.

CUSTODY SEAL

This package confirms to the conditions
and limitations specified in 49 CFR 173.4

Date Sealed: _____

Sealed By: _____

GCAL-17

Technical Memorandums for SWMUs 54 and 55



TECHNICAL MEMORANDUM

Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 54, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/NAVFAC SE
Tim Gordon/USEPA
Wilmarie Rivera/PREQB

COPIES: David Criswell/NAVFAC SE
Pedro Ruiz/NAPR
Mark Kimes/Baker

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: October 22, 2009

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Naval Facilities Engineering Command Southeast (NAVFAC SE) to conduct a pilot study at Solid Waste Management Unit (SWMU) 54 located at Naval Activity Puerto Rico (NAPR), formerly known as Naval Station Roosevelt Roads, Ceiba, Puerto Rico. This work is being performed under Contract Number N62470-08-D-1006, Task Order JM04. As detailed in the *Pilot Study Work Plan for SWMU 54* (AGVIQ-CH2M HILL, 2009), the pilot study will evaluate the effectiveness of in situ bioremediation (ISB) in remediating groundwater contaminated with trichloroethene (TCE) and benzene and reducing the time required to achieve corrective action objectives. The pilot study will be performed on the TCE and benzene plumes identified in the *Final Corrective Measures Study (CMS) Final Report* (Baker Environmental, Inc. [Baker], 2005).

As described in the *Pilot Study Work Plan*, ISB pilot testing at SWMU 54 will include a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, ISB injections, and performance monitoring (AGVIQ-CH2M HILL, 2009). Installation and sampling of monitoring wells has been completed as described in the Work Plan; however, the analytical results indicate the extents of the TCE and benzene plumes have not been fully characterized. This technical memorandum presents the August 2009 analytical results and makes recommendations for additional site work based on the evaluation of these data.

Baseline Sampling Event Results

The baseline sampling event at SWMU 54 was conducted in August 2009. Sampling was performed to verify the current concentrations of TCE and benzene in groundwater and determine any changes in the locations or extents of the contaminant plumes since the last sampling event in 2002. During the August event, the following work was performed:

- Installed six new monitoring wells (54MW01, 54MW02, 54MW03, 54MW04, 54MW05, and 54MW06) in the benzene plume area and collected samples from these wells for the analysis of benzene using U.S. Environmental Protection Agency (EPA) Method 8260B.
- Installed nine new monitoring wells (54MW07, 54MW08, 54MW09, 54MW10, 54MW11, 54MW12, 54MW13, 54MW14, and 510MW5R) in the TCE plume area and collected samples from these wells for the analysis of TCE using EPA Method 8260B.

Well locations are illustrated on Figure 1.

TCE Plume

The TCE sampling results obtained in August 2009 are summarized in Table 1. Table 1 also includes the TCE sampling results from February 2002 (Baker, 2005). Figures 2 and 3 depict the distribution of TCE in groundwater in 2002 and 2009, respectively. A comparison of the 2002 TCE distribution (Figure 2) with the 2009 TCE distribution (Figure 3) suggests that TCE concentrations in groundwater have decreased since 2002. However, the 2009 data show the highest TCE concentration (139 micrograms per liter [$\mu\text{g}/\text{L}$]) now occurs in 54MW08, which is located about 50 feet south of 510MW5 where the highest TCE concentration occurred in 2002. The 2009 data also show that the extent of the TCE plume to the southeast, west, and south, which includes the area with the highest measured TCE concentration in the vicinity of 54MW08, has not been fully characterized.

TABLE 1
 Summary of 2002 and 2009 TCE Analytical Results

Well Identification	Sample Date	TCE Concentration ($\mu\text{g}/\text{L}$)
54MW07	08/2009	72.6
54MW08	08/2009	139
54MW09	08/2009	42.5
54MW10	08/2009	29.6
54MW11	08/2009	35.7
54MW12	08/2009	18.8
54MW13	08/2009	2.5
54MW14	08/2009	6.18
510MW05R	08/2009	50.9
54TW03 ^a	02/2002	53
54TW04 ^a	02/2002	84

TABLE 1
 Summary of 2002 and 2009 TCE Analytical Results

Well Identification	Sample Date	TCE Concentration (µg/L)
54TW05 ^a	02/2002	170D
54TW06 ^a	02/2002	34
54TW11 ^a	02/2002	140
54TW12 ^a	02/2002	5U
54TW14 ^a	02/2002	25
54TW18 ^a	02/2002	5U
54PZ01 ^a	02/2002	2.4J
510MW5 ^a	02/2002	190

Notes:

^a *Final Corrective Measures Study Final Report for SWMUs 54 and 55* (Baker, 2005)

D = The sample was diluted for analysis.

J = The analyte was detected between the laboratory minimum detection limit and reporting limit.

µg/L = microgram per liter

U = The analyte was not detected above the laboratory reporting limit.

Benzene Plume

The benzene sampling results obtained in August 2009 are summarized in Table 2. Table 2 also includes benzene sampling results from February 2002 (Baker, 2005). Figures 4 and 5 depict the distribution of benzene in groundwater in 2002 and 2009, respectively. A comparison of the 2002 and 2009 data indicates benzene was detected at significantly higher concentrations during the 2009 sampling event than during the 2002 event (9,260 µg/L in 54MW06 versus 3,000 µg/L in 54TW15). In addition, benzene was detected in excess of its treatment standard at multiple locations in 2009. These results indicate the potential benzene source area has not been identified, and the extent of benzene in groundwater has not been defined.

TABLE 2
 Summary of 2002 and 2009 Benzene Analytical Results

Well Identification	Sample Date	Benzene Concentration (µg/L)
54MW01	08/2009	707
54MW02	08/2009	394
54MW03	08/2009	347
54MW04	08/2009	2.83
54MW05	08/2009	2.2
54MW06	08/2009	9,260
54TW15 ^a	02/2002	3,000

Notes:

^a *Final Corrective Measures Study Final Report for SWMUs 54 and 55* (Baker, 2005)

Conclusions and Recommendations

Test results from the 2009 sampling indicate that the extent of TCE and benzene in groundwater beneath SWMU 54 has not been fully defined, and additional site characterization is necessary to determine the extent of dissolve contamination and identify the individual source areas. Based on this information, CH2M HILL recommends the following:

- Shift the TCE pilot study injection wells to the locations shown on Figure 3.
- Install four monitoring wells at the locations shown on Figure 3 to delineate the extent of TCE beneath SWMU 54. Once the wells are installed and developed, collect groundwater samples for the analyses of total volatile organic compounds (VOCs), dissolved iron, dissolved manganese, sulfate, sulfide, total organic carbon, methane, ethene, ethane, and alkalinity using the analytical methods required in the *Pilot Study Work Plan for SWMU 54* (AGVIQ-CH2M HILL, 2009).
- Delay the ISB pilot test on the benzene plume until the source area and plume extent are better characterized and the cost and feasibility of ISB using ORC can be properly assessed.
- Collect groundwater samples from previously installed monitoring wells 510MW1, 510DW1, 510MW2, 510DW2, 510MW3, and 510MW4 (see Figure 5), and analyze for VOCs using EPA Method 8260B.
- Install five monitoring well pairs at the locations shown on Figure 5 to delineate the extent of benzene beneath SWMU 54. The proposed well pairs will consist of two screened intervals, 5 to 15 feet below ground surface (ft bgs) and 15 to 25 ft bgs. Once installed and developed, collect groundwater samples from these wells for the analysis of VOCs using EPA Method 8260B.
- Survey the locations and elevations of the newly installed wells and develop a potentiometric map.
- Evaluate the data to determine the most suitable course of corrective action for the site. For the TCE plume, ISB using enhanced reductive dechlorination is still the best remediation option and a pilot test should be completed.

Once additional characterization of the benzene plume is complete, CH2M HILL will evaluate ISB and alternative technologies for source and plume remediation. Remedial technologies may include excavation, an air sparging trench, and ISB using ORC.

References

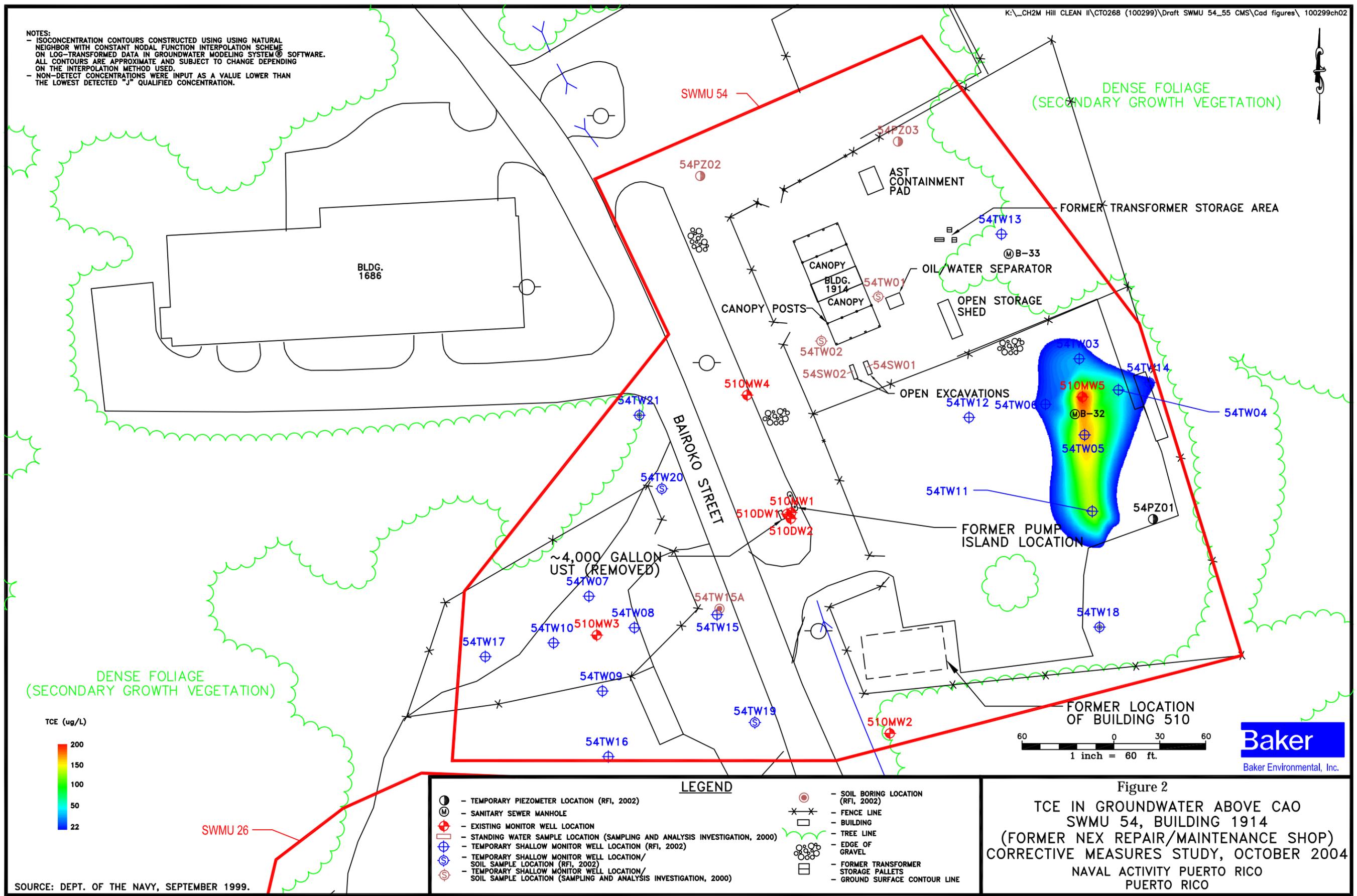
- AGVIQ-CH2M HILL. 2009. *Pilot Study Work Plan for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. January.
- Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figures



FIGURE 1
 Site Location Map
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico

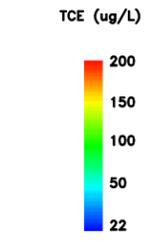
NOTES:
 - ISOCONCENTRATION CONTOURS CONSTRUCTED USING USING NATURAL NEIGHBOR WITH CONSTANT NODAL FUNCTION INTERPOLATION SCHEME ON LOG-TRANSFORMED DATA IN GROUNDWATER MODELING SYSTEM® SOFTWARE. ALL CONTOURS ARE APPROXIMATE AND SUBJECT TO CHANGE DEPENDING ON THE INTERPOLATION METHOD USED.
 - NON-DETECT CONCENTRATIONS WERE INPUT AS A VALUE LOWER THAN THE LOWEST DETECTED "J" QUALIFIED CONCENTRATION.



DENSE FOLIAGE (SECONDARY GROWTH VEGETATION)

60 0 30 60
 1 inch = 60 ft.

Baker
 Baker Environmental, Inc.

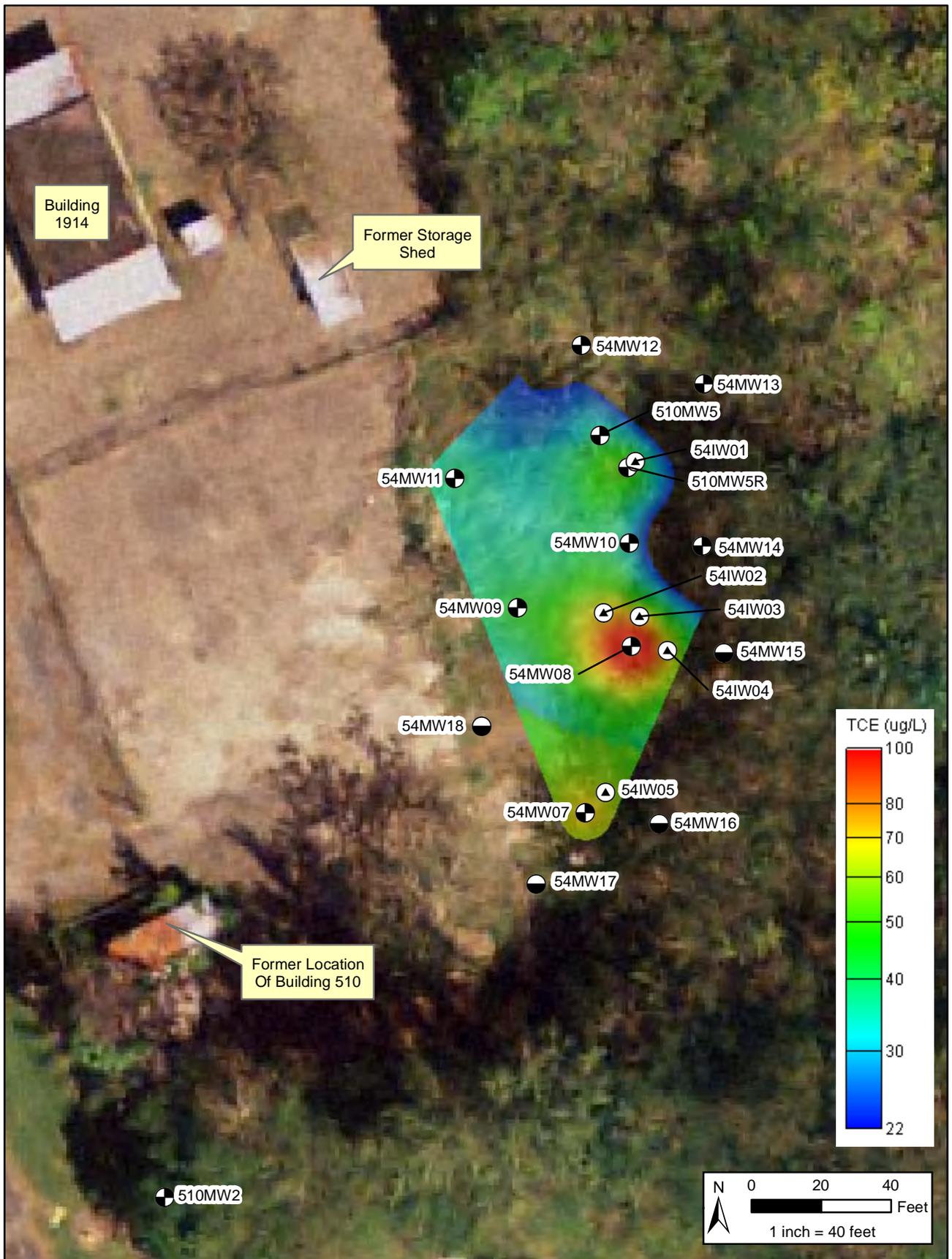


LEGEND

⊙	- TEMPORARY PIEZOMETER LOCATION (RFI, 2002)	⊙	- SOIL BORING LOCATION (RFI, 2002)
Ⓜ	- SANITARY SEWER MANHOLE	⊗	- FENCE LINE
⊕	- EXISTING MONITOR WELL LOCATION	▭	- BUILDING
⊕	- STANDING WATER SAMPLE LOCATION (SAMPLING AND ANALYSIS INVESTIGATION, 2000)	⊕	- TREE LINE
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION (RFI, 2002)	⊕	- EDGE OF GRAVEL
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION / SOIL SAMPLE LOCATION (RFI, 2002)	⊕	- FORMER TRANSFORMER STORAGE PALLETS
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION / SOIL SAMPLE LOCATION (SAMPLING AND ANALYSIS INVESTIGATION, 2000)	—	- GROUND SURFACE CONTOUR LINE

Figure 2
 TCE IN GROUNDWATER ABOVE CAO
 SWMU 54, BUILDING 1914
 (FORMER NEX REPAIR/MAINTENANCE SHOP)
 CORRECTIVE MEASURES STUDY, OCTOBER 2004
 NAVAL ACTIVITY PUERTO RICO
 PUERTO RICO

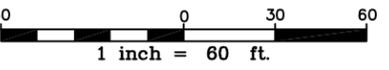
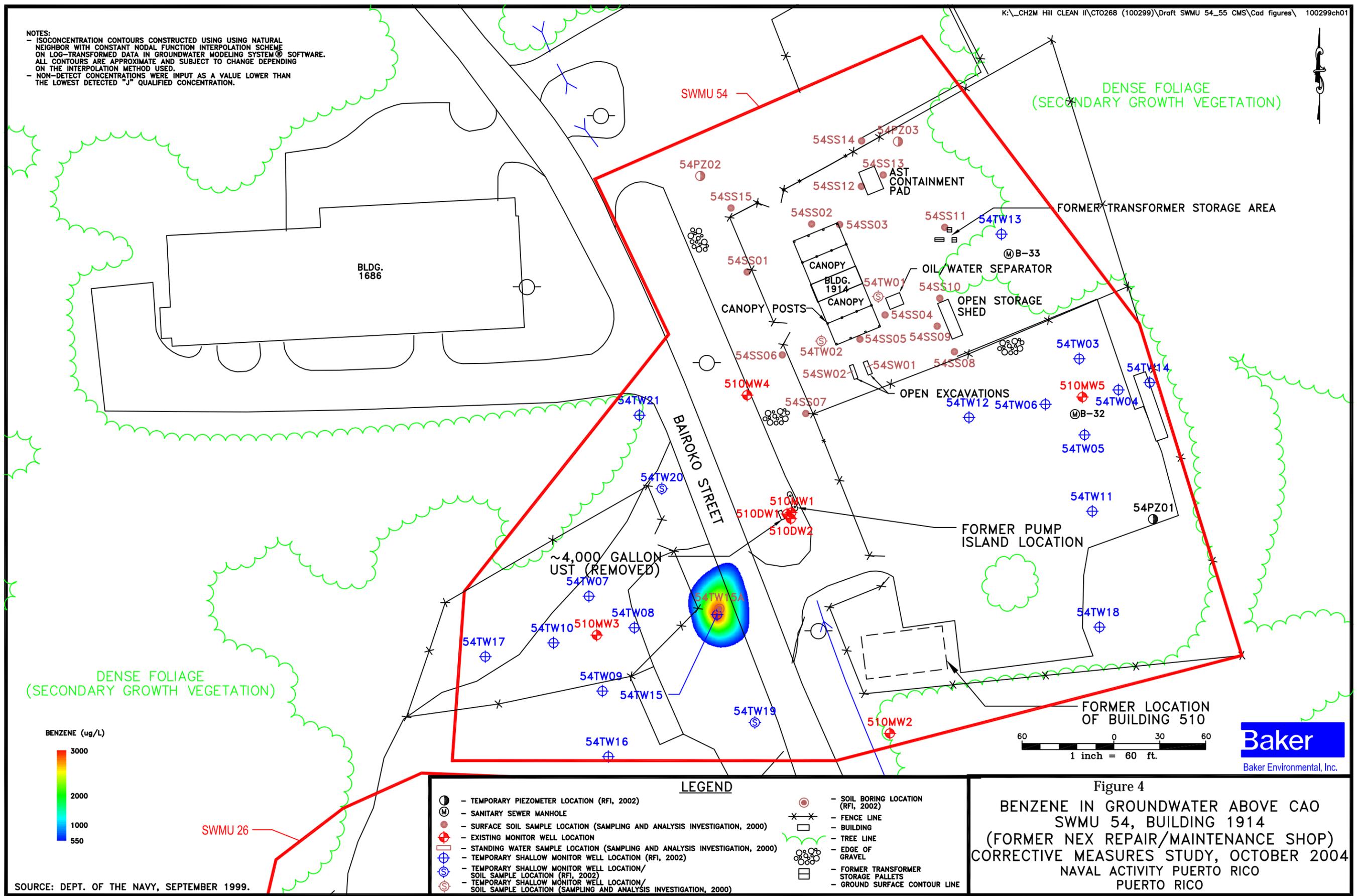
SOURCE: DEPT. OF THE NAVY, SEPTEMBER 1999.



- ⊕ Monitoring Well Location
- ⊖ Proposed Monitoring Well Location
- ⊕ Proposed Injection Well Location
- Former Structure

FIGURE 3
 Distribution Of TCE In Groundwater, August 2009
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico

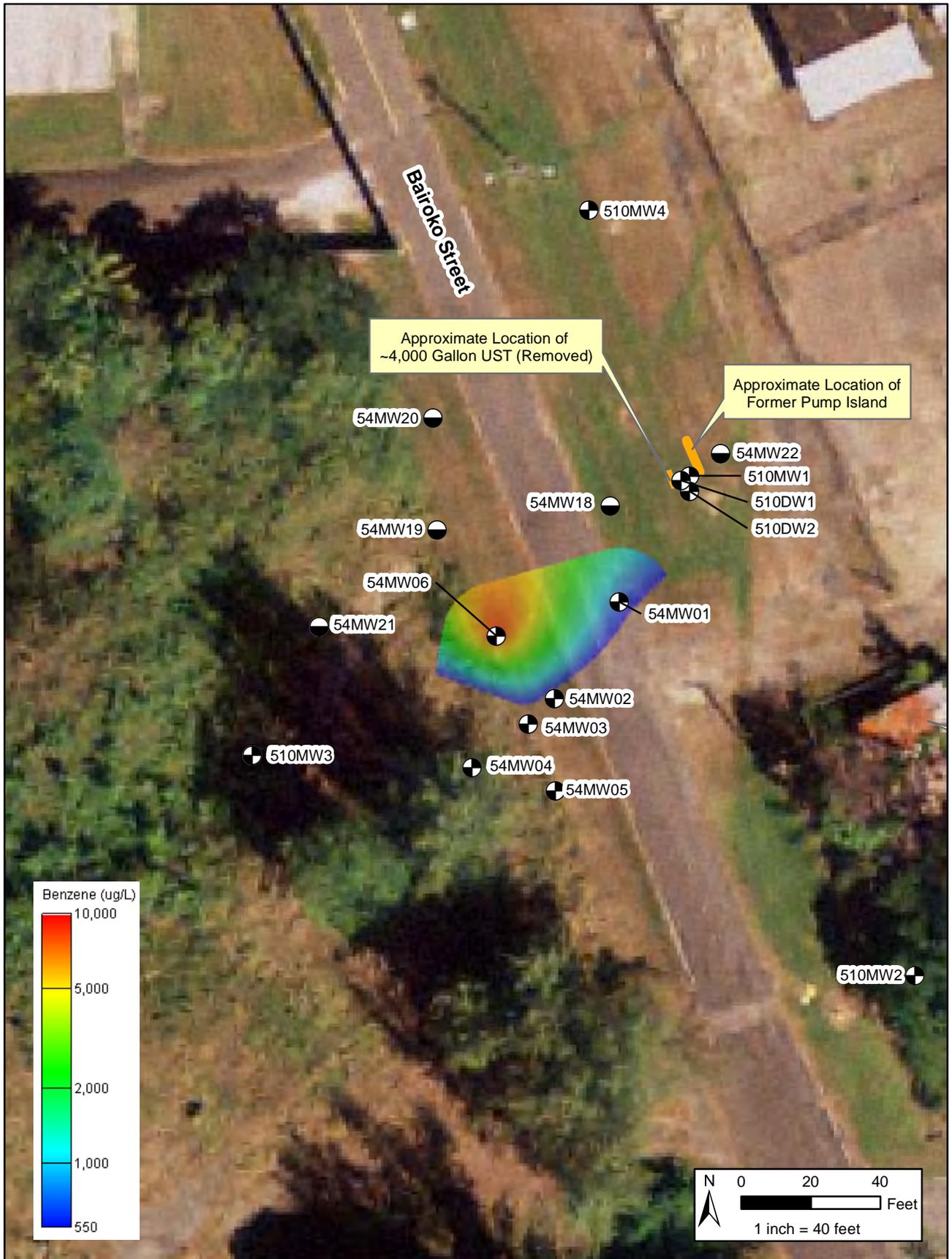
NOTES:
 - ISOCONCENTRATION CONTOURS CONSTRUCTED USING USING NATURAL NEIGHBOR WITH CONSTANT NODAL FUNCTION INTERPOLATION SCHEME ON LOG-TRANSFORMED DATA IN GROUNDWATER MODELING SYSTEM® SOFTWARE. ALL CONTOURS ARE APPROXIMATE AND SUBJECT TO CHANGE DEPENDING ON THE INTERPOLATION METHOD USED.
 - NON-DETECT CONCENTRATIONS WERE INPUT AS A VALUE LOWER THAN THE LOWEST DETECTED "J" QUALIFIED CONCENTRATION.



LEGEND	
⊙	- TEMPORARY PIEZOMETER LOCATION (RFI, 2002)
Ⓜ	- SANITARY SEWER MANHOLE
●	- SURFACE SOIL SAMPLE LOCATION (SAMPLING AND ANALYSIS INVESTIGATION, 2000)
⊕	- EXISTING MONITOR WELL LOCATION
⊕	- STANDING WATER SAMPLE LOCATION (SAMPLING AND ANALYSIS INVESTIGATION, 2000)
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION (RFI, 2002)
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION / SOIL SAMPLE LOCATION (RFI, 2002)
⊕	- TEMPORARY SHALLOW MONITOR WELL LOCATION / SOIL SAMPLE LOCATION (SAMPLING AND ANALYSIS INVESTIGATION, 2000)
⊙	- SOIL BORING LOCATION (RFI, 2002)
✕	- FENCE LINE
□	- BUILDING
—	- TREE LINE
⊖	- EDGE OF GRAVEL
⊖	- FORMER TRANSFORMER STORAGE PALLETS
—	- GROUND SURFACE CONTOUR LINE

Figure 4
 BENZENE IN GROUNDWATER ABOVE CAO
 SWMU 54, BUILDING 1914
 (FORMER NEX REPAIR/MAINTENANCE SHOP)
 CORRECTIVE MEASURES STUDY, OCTOBER 2004
 NAVAL ACTIVITY PUERTO RICO
 PUERTO RICO

SOURCE: DEPT. OF THE NAVY, SEPTEMBER 1999.



Bairko Street

Approximate Location of ~4,000 Gallon UST (Removed)

Approximate Location of Former Pump Island

510MW4

54MW20

54MW22

54MW18

510MW1

510DW1

510DW2

54MW19

54MW06

54MW01

54MW21

54MW02

54MW03

510MW3

54MW04

54MW05

510MW2

FIGURE 5
 Distribution of Benzene In Groundwater, August 2009
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico



TECHNICAL MEMORANDUM

Phase II Additional Sampling Requirements for SWMU 54, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/PMO SE

COPIES: David Criswell/PMO SE
Pedro Ruiz/NAPR

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: February 22, 2010

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Base Closure and Realignment (BRAC) Program Management Office Southeast (PMO SE) to conduct a pilot study at Solid Waste Management Unit (SWMU) 54 located at Naval Activity Puerto Rico (NAPR), formerly known as Naval Station Roosevelt Roads, Ceiba, Puerto Rico. This work is being performed under Contract No. N62470-08-D-1006, Task Order JM04. As detailed in the *Pilot Study Work Plan for SWMU 54* (AGVIQ-CH2M HILL, 2009a), the purpose of the pilot study is to evaluate the effectiveness of in situ bioremediation (ISB) to remediate groundwater contaminated with trichloroethene (TCE) and benzene. The pilot study is being performed on the TCE and benzene plumes identified in the *Final Corrective Measures Study (CMS) Final Report* (Baker Environmental, Inc. [Baker], 2005).

As described in the Work Plan, ISB pilot testing at SWMU 54 includes a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, ISB injections, and performance monitoring (AGVIQ-CH2M HILL, 2009a). Installation and sampling of monitoring wells has been completed as described in the Work Plan, as well as the ISB injection at the TCE plume. In addition, 4 monitoring wells at the TCE plume and 10 monitoring wells at the benzene plume were installed and sampled, according to the *Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 54* (AGVIQ-CH2M HILL, 2009b). Results from this sampling event indicate that the extent of TCE contamination has been essentially delineated; however, the benzene area has not been fully characterized, vertically or horizontally.

This technical memorandum presents the December 2009 and January 2010 analytical results and makes recommendations for additional site work based on the evaluation of these data.

Phase I Additional Characterization Sampling Event Results

The phase I additional characterization sampling event was conducted between December 2009 and January 2010. Sampling was performed to supplement the August 2009 groundwater monitoring data and further characterize the TCE and benzene plumes. During the Phase I event, the following work was performed:

- Installed four new monitoring wells (54MW15, 54MW16, 54MW17, and 54MW18) in the TCE plume area and collected samples from these wells for the analysis of volatile organic compounds (VOCs) using U.S. Environmental Protection Agency (EPA) Method 8260B.
- Installed five new injection wells (54IW01, 54IW02, 54IW03, 54IW04, and 54IW05) in the TCE plume area and collected samples from these wells for the analysis of VOCs using EPA Method 8260B.
- Installed 10 new monitoring wells (54MW19, 54MW20, 54MW21, 54MW22, 54MW23, 54MW24, 54MW25, 54MW26, 54MW27, and 54MW28) in the benzene plume area and collected samples from these wells for the analysis of VOCs using EPA Method 8260B.
- Sampled existing monitoring wells (54MW01, 54MW02, 54MW03, 54MW04, 54MW05, and 54MW06) for the analysis of VOCs using EPA Method 8260B.

Well locations are illustrated on Figure 1.

TCE Plume

The TCE sampling results from August and December 2009 are summarized in Table 1. Figure 2 depicts the distribution of TCE in groundwater using the August and December data collectively. TCE concentrations were detected slightly above the NAPR cleanup criterion of 22 micrograms per liter ($\mu\text{g/L}$) at wells 54MW11 (35.7 $\mu\text{g/L}$), 54MW15 (39.2 $\mu\text{g/L}$), 54MW16 (26.3 $\mu\text{g/L}$), and 54MW18 (26.7 $\mu\text{g/L}$), indicating that the extent of TCE in groundwater exceeding the cleanup criterion has been essentially defined in TCE plume area of SWMU 54.

TABLE 1
 Summary of August and December 2009 TCE Analytical Results

Well Identification	Sample Date	TCE Concentration ($\mu\text{g/L}$)
54MW07	08/2009	72.6
54MW08	08/2009	139
54MW09	08/2009	42.5
54MW10	08/2009	29.6
54MW11	08/2009	35.7
54MW12	08/2009	18.8
54MW13	08/2009	2.5
54MW14	08/2009	6.18
54MW15	12/2009	39.2

TABLE 1
 Summary of August and December 2009 TCE Analytical Results

Well Identification	Sample Date	TCE Concentration (µg/L)
54MW16	12/2009	26.3
54MW17	12/2009	7.96
54MW18	12/2009	26.7
54IW01	12/2009	55.2
54IW02	12/2009	246
54IW03	12/2009	181
54IW04	12/2009	256
54IW05	12/2009	75.6

Notes:

Bold indicates the measured concentration exceeds the TCE cleanup criterion of 22 µg/L.

In addition to TCE, the VOCs 1,2-dichlorobenzene, cis-1,2-dichloroethene (DCE), chloroform, and methylene chloride were also detected. However, individual constituent concentrations were less than 10 µg/L and do not warrant further investigation.

Benzene Plume

The benzene sampling results from August 2009 and January 2010 are summarized in Table 2. Figures 3 and 4 depict the distribution of benzene in the deep and shallow zones of the overburden aquifer, respectively. Two separate areas of benzene contamination exceeding the 550 µg/L cleanup criterion were identified: one immediately south of the former pump island and 4,000-gallon underground storage tank (UST) (Figure 3) and the second west of Bairoko Street (Figure 4). It is likely the benzene detected west of Bairoko Street is associated with a tank truck fuel spill, rather than the UST. The benzene near well 54MW06 (west of Bairoko Street) appears to occur primarily in the shallow zone (5 to 15 feet below ground surface [ft bgs]), while the benzene near well 54MW27 (south of the former pump island) appears to exist in a deeper zone (25 to 40 ft bgs). The data on Figures 3 and 4 demonstrate that the lateral and vertical extent of benzene contamination has not been fully defined in either area.

TABLE 2
 Summary of August 2009 and January 2010 Benzene Analytical Results

Well Identification	Sample Date	Benzene Concentration (µg/L)
54MW01	08/2009	707
54MW02	08/2009	394
54MW03	08/2009	347
54MW04	08/2009	2.83
54MW05	08/2009	2.2

TABLE 2
 Summary of August 2009 and January 2010 Benzene Analytical Results

Well Identification	Sample Date	Benzene Concentration (µg/L)
54MW06	08/2009	9,260
54MW01	01/2010	653
54MW02	01/2010	500
54MW03	01/2010	357
54MW04	01/2010	<5
54MW05	01/2010	1.52
54MW06	01/2010	14,200
54MW19	01/2010	45.2
54MW20	01/2010	208
54MW21	01/2010	15.2
54MW22	01/2010	20.7
54MW23	01/2010	<5
54MW24	01/2010	<5
54MW25	01/2010	<5
54MW26	01/2010	<5
54MW27	01/2010	7,410
54MW28	01/2010	<5

Notes:

Bold indicates the measured concentration exceeds the benzene cleanup criterion of 550 µg/L.

In addition to benzene, the VOCs acetone, chloroform, cis-1,2-DCE, cyclohexane, 1,2-dichloroethane (DCA), ethylbenzene, 4-methyl-2-pentanone, isopropylbenzene, methylcyclohexane, methyl tert butyl ether, toluene, TCE, and xylenes were detected in benzene plume wells during this sampling event. With the exception of concentrations of 1,2-DCA (8.04 µg/L) at 54MW02, ethylbenzene at 54MW01 (893 µg/L), TCE at 54MW04 (24.9 µg/L) and 54MW05 (40.9 µg/L), and ethylbenzene (1,300 µg/L) and toluene (1,150 µg/L) at 54MW06, the VOCs were either measured below their Maximum Contaminant Levels or did not exceed the previously calculated risk-based cleanup criteria (Baker, 2005). Therefore, these detections do not warrant further investigation. However, the two TCE detections at wells 54MW04 and 54MW05 exceeded the cleanup criterion of 22 µg/L. These wells are located west of Bairoko Street and are separated by wells with lower TCE concentrations, suggesting a separate TCE source may be present. In addition, neither of these wells had benzene detections, further suggesting the existence of a separate TCE source.

Conclusions and Recommendations

Test results from the 2009 and 2010 sampling events indicate that the extent of TCE contamination in the known source area, as established by Baker in the CMS, has been

adequately characterized and no additional groundwater monitoring wells are required for this purpose. However, data associated with the former fueling system indicate that the vertical and horizontal extent of benzene in the overburden aquifer has not been determined. The data also show a second benzene source area west of Bairoko Street. Therefore, additional site characterization is necessary to determine the horizontal and vertical extent of benzene in groundwater.

During sampling associated with the benzene plume, TCE was detected above the cleanup criterion of 22 µg/L in monitoring wells 54MW04 and 54MW05. These wells are located west of Bairoko Street and appear to represent a separate area of TCE contamination; therefore, the installation of one additional monitoring well is recommended to determine if a separate TCE source exists.

Based on these findings, CH2M HILL recommends the following:

- TCE Plume: Return to original limited VOC analyte list (TCE, cis-1,2-DCE, and vinyl chloride) for remainder of groundwater sampling at the TCE area.
- Benzene Plume: Return to original analyte list (benzene only) for remainder of groundwater sampling at the benzene area.
- Benzene Plume (Deep): Install eight monitoring wells at the locations shown on Figure 3 to delineate the extent of benzene in the deep zone beneath SWMU 54. The new monitoring wells will include deep wells (screened 25 to 40 ft bgs) paired with 54MW01 and 54MW27 to ensure the benzene contamination in this area is confined to this zone. Well pairs (screened 15 to 25 and 25 to 40 feet bgs) will be installed around 54MW27 to fully define the extent of benzene contamination in this area. Once the wells are installed and developed, collect groundwater samples for the analyses of benzene using EPA Method 8260B.
- Benzene Plume (Shallow): Install four monitoring wells at the locations shown on Figure 4 to delineate the extent of benzene in the shallow zone beneath SWMU 54. The new monitoring wells will include deep wells (screened 15 to 25 ft bgs) paired with 54MW06 and 54MW02 to ensure the benzene contamination in this area is only in the shallow zone and a well pair between 54MW06 and 510MW3 (screened 5 to 15 ft bgs and 15 to 25 ft bgs) to further define the extent of benzene contamination in this direction and limit the area requiring treatment. Once the wells are installed and developed, collect groundwater samples for the analyses of benzene using EPA Method 8260B.
- Benzene Plume (Shallow): Install one monitoring well (screened 5 to 15 ft bgs) east of 54MW04 and 54MW05 at the location shown on Figure 4 to determine if the TCE detections are linked to the TCE plume east of Bairoko Street. Once the well is installed and developed, collect groundwater samples for the analyses of TCE using EPA Method 8260B.
- Survey the locations and elevations of the newly installed wells.
- Evaluate the data to determine the most suitable course of corrective action for the benzene plume.

Once additional characterization of the benzene plume is complete, CH2M HILL will evaluate ISB and alternative technologies for source and plume remediation. Remedial technologies may include excavation, an air sparging trench, and ISB using oxygen release compound (ORC®).

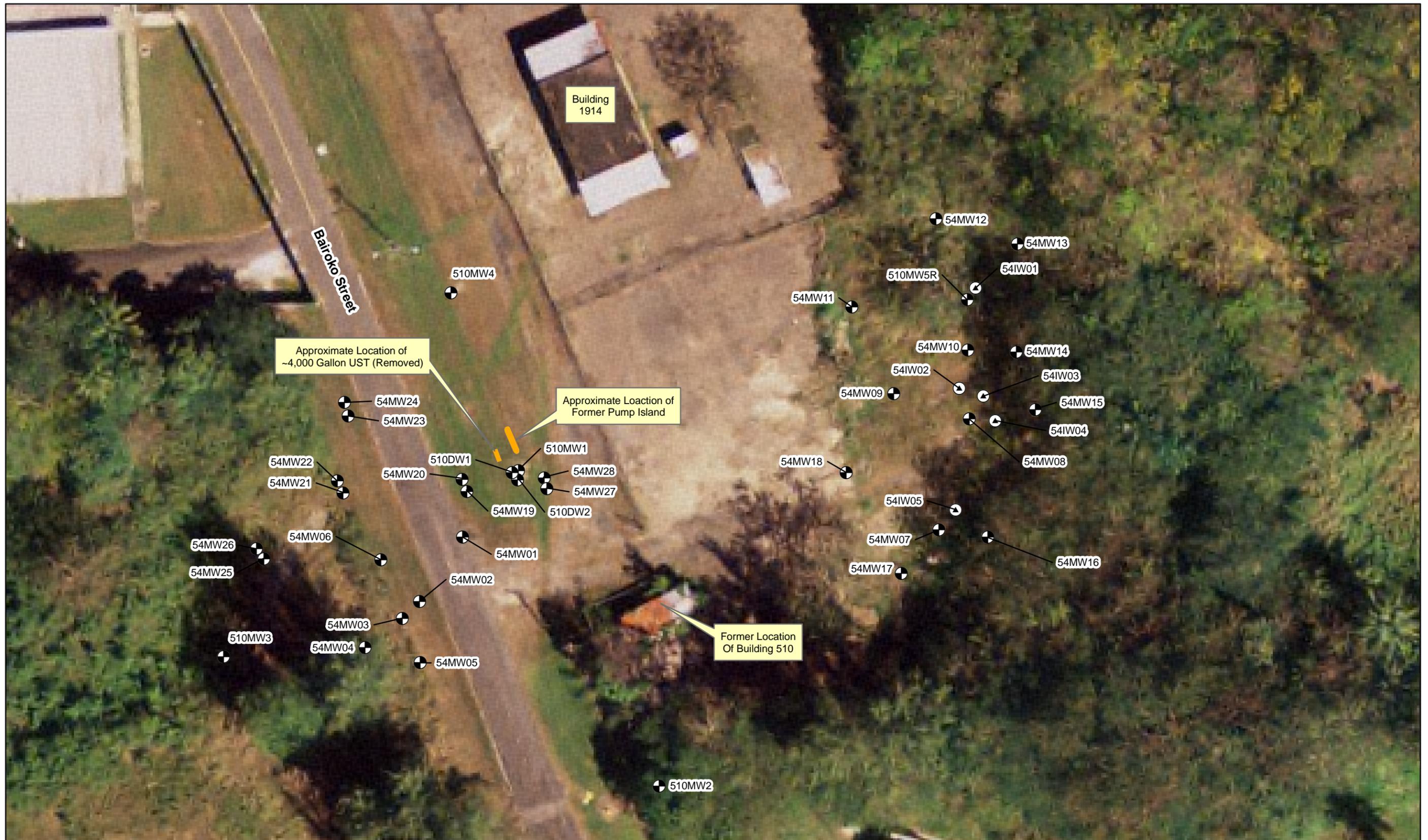
References

AGVIQ-CH2M HILL. 2009a. *Pilot Study Work Plan for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. January.

AGVIQ-CH2M HILL. 2009b. *Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. October.

Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figures



-  Monitoring Well Location
-  Injection Well Location
-  Former Structure

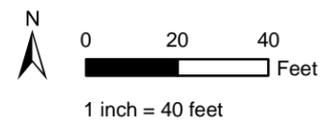
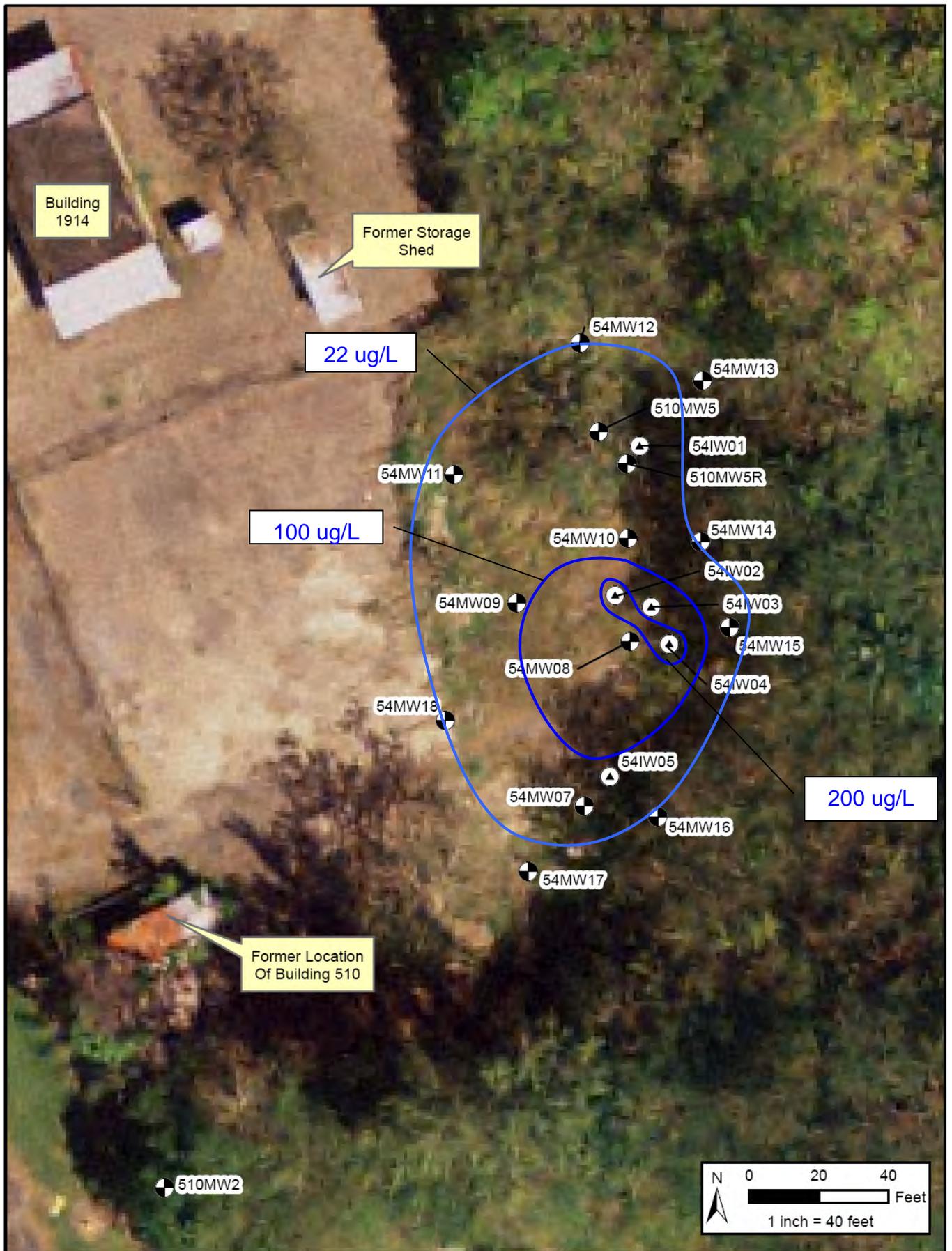
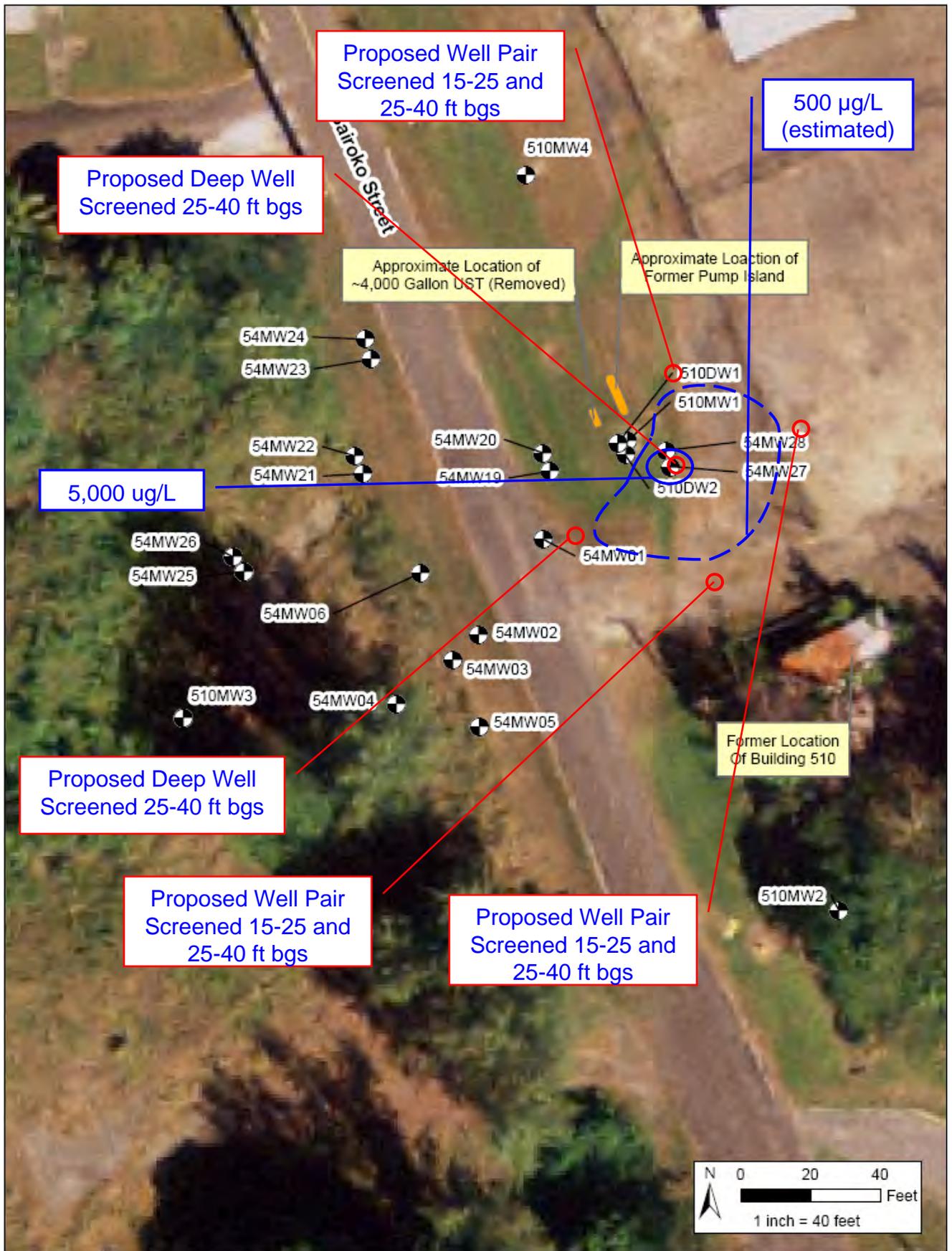


FIGURE 1
 Site Location Map
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico



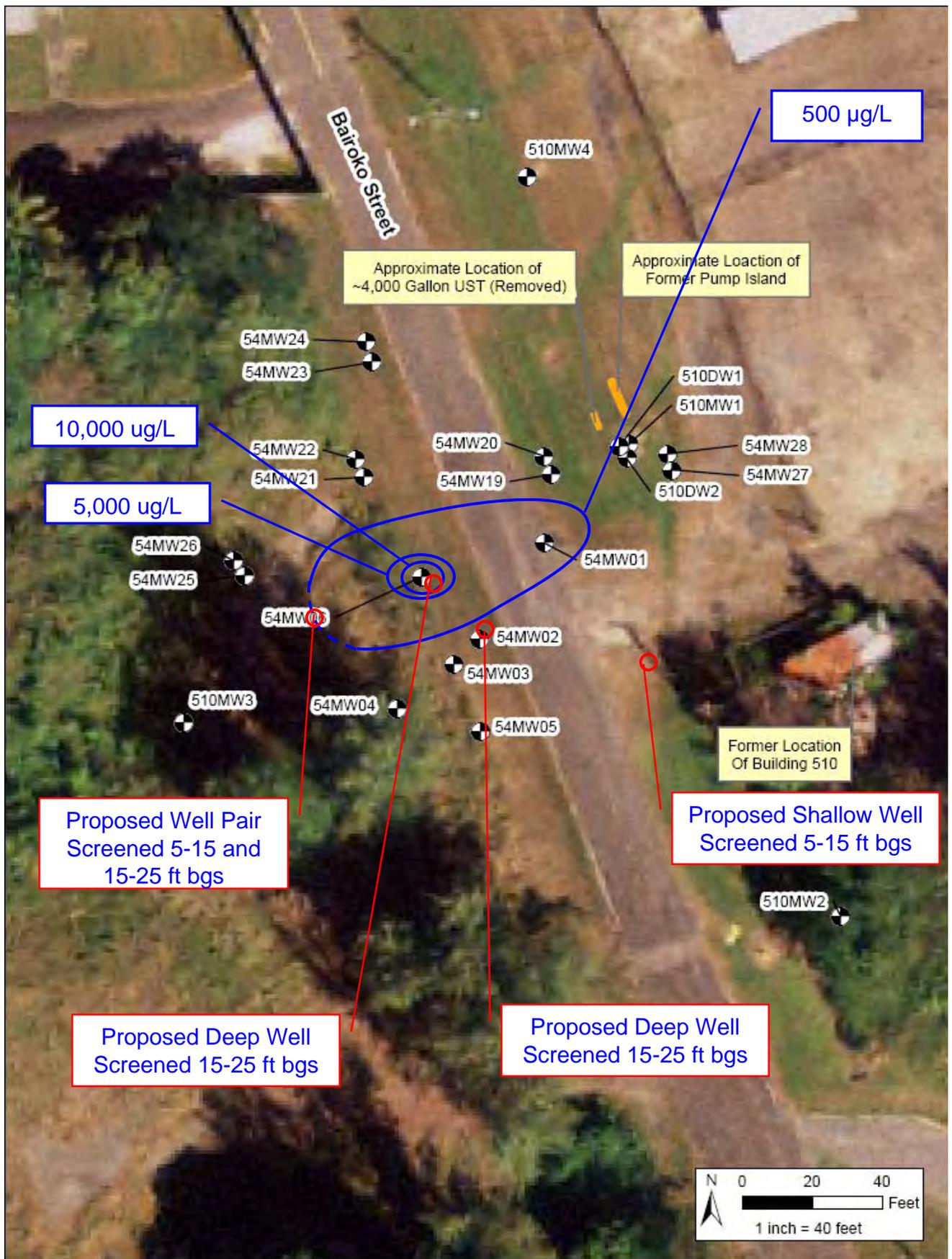
-  Monitoring Well Location
-  Injection Well Location

FIGURE 2
 Distribution Of TCE In Groundwater
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico



Monitoring Well Location

FIGURE 3
 Distribution of Benzene In Groundwater - Deep Zone
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico



Monitoring Well Location

FIGURE 4
 Distribution of Benzene In Groundwater - Shallow Zone
 SWMU 54
 Naval Station Roosevelt Roads, Puerto Rico



TECHNICAL MEMORANDUM

Phase III Additional Sampling Requirements for SWMU 54, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/PMO SE
Pedro Ruiz/NAPR

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: July 29, 2010

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) was retained by the Department of the Navy, Base Closure and Realignment (BRAC) Program Management Office Southeast (PMO SE) to conduct pilot testing at Solid Waste Management Unit (SWMU) 54 located at Naval Activity Puerto Rico (NAPR), formerly known as Naval Station Roosevelt Roads (NSRR), Ceiba, Puerto Rico. This work was performed under Contract No. N62470-08-D-1006, Task Order JM04. This technical memorandum (TM) presents the results obtained following the collection of water quality samples in April 2010 from wells that were recently (February and March 2010) installed to determine the horizontal and vertical extent of benzene in groundwater and to determine if trichloroethene (TCE) was present downgradient of the previously identified TCE source area. The TM also includes recommendations for additional work based on interpretation of the recently collected data.

Background

In January 2009, AGVIQ-CH2M HILL prepared and submitted a work plan to the Navy, describing the procedures that would be used to evaluate the effectiveness of in situ bioremediation (ISB) to remediate groundwater impacted by TCE and benzene beneath SWMU 54 (AGVIQ-CH2M HILL, 2009a). The areas targeted for pilot testing were based on the TCE and benzene plume maps presented in the *Final Corrective Measures Study [CMS] Final Report* (Baker Environmental, Inc. [Baker], 2005).

The work included the installation of 14 baseline monitoring wells (510MWR and 54MW01 through 54MW13), collection of baseline water quality samples, installation of five ISB injection wells (54IW01 through 54IW05), installation of five additional monitoring wells (54MW14 through 54MW18) to define the extent of TCE following review of the baseline data, completion of an ISB injection event, and completion of two quarterly post-injection groundwater sampling events. Sampling results indicated the extent of TCE was determined; however, the horizontal and vertical extent of the benzene plume was not defined. Therefore, in October 2009, AGVIQ-CH2M HILL submitted a revised work plan to

the Navy, describing the scope of work to install 10 additional monitoring wells (54MW19 through 54MW28) to determine the vertical and horizontal extent of benzene in groundwater (AGVIQ-CH2M HILL, 2009b). Between October and December 2009, monitoring wells 54MW19 through 54MW28 were installed and sampled. Sampling results from these wells suggested that the benzene plume appeared to be split into two separate sources: a deep source associated with the former underground storage tanks (USTs) located on the east side of Bairoko Street, and a shallow source related to a surface spill of fuel that reportedly occurred on the west side of Bairoko Street (Figure 1).

Based on these results, in February 2010, AGVIQ-CH2M HILL prepared a TM, recommending the installation of 12 additional monitoring wells (54MW29 through 54MW33 and 54MW35 through 54MW41) to determine the extent of benzene in groundwater (AGVIQ-CH2M HILL, 2010). In addition, a thirteenth well (54MW34) was proposed to determine if TCE was present in groundwater along the southern portion of SWMU 54 because TCE was detected in previously installed well 54MW18.

Therefore, between late-February and early-April 2010, AGVIQ-CH2M HILL installed and collected groundwater samples from monitoring wells 54MW29 through 54MW41 as part of the Phase II investigation. The sampling results obtained from the Phase II work are discussed in the following subsections, and well locations from the recent Phase II work, as well as the previous work, are provided on Figure 1.

Phase II Additional Characterization Sampling Results

The Phase II sampling event was conducted on April 14 and 15, 2010. Sampling results from this event are summarized in Table 1, along with previous data collected in September 2009, December 2009, and January 2010. Isoconcentration maps depicting the April 2010 extent of benzene to the corrective action objective (CAO) of 550 micrograms per liter ($\mu\text{g}/\text{L}$) in the shallow zone (upper 5 to 15 feet of the water table aquifer) and deep zone (lower 15 to 25 feet of the water table aquifer) are illustrated on Figures 2 and 3, respectively. In addition, TCE results from well 54MW34 are also discussed below.

Distribution of Benzene

The April 2010 sampling results confirm that there are two benzene plumes: one existing in the shallow zone primarily on the west side of Bairoko Street and one in the deep zone on the east side of Bairoko Street (refer to Figures 2 and 3). The data also identified a previously unknown area of shallow benzene contamination that is present in 54MW34 located on the southeast side of Bairoko Street near a drainage ditch. Sampling results by zone are discussed below.

Shallow Zone (5 to 15 feet)

Sampling results from groundwater wells installed on the west side of Bairoko Street (54MW38, 54MW39, and 54MW41) indicate that benzene concentrations above the CAO are confined to the shallow zone (5 to 15 feet) of the water table aquifer and that the horizontal extent of benzene has been defined to the CAO (Figure 2). However, the sampling results also indicate that a benzene concentration of 10,800 $\mu\text{g}/\text{L}$ was detected in shallow zone monitoring well 54MW34 located on the east-side of Bairoko Street. Monitoring well 54MW34 is located approximately 60 feet southeast of monitoring well 54MW06, where a benzene concentration of 14,200 $\mu\text{g}/\text{L}$ was measured in January 2010. Because well 54MW34

is also separated by shallow zone wells 54MW02 and 54MW03 that have significantly lower benzene concentrations, it is likely that the benzene detected in 54MW34 is unrelated to the surface spill of fuel that reportedly occurred on the west side of Bairoko Street.

Sampling results from wells installed east of Bairoko Street indicate that benzene concentrations in the upper 15 feet of the water table aquifer are below the CAO (Figure 2).

Deeper Zone (15 to 25 feet)

As shown on Figure 3, benzene concentrations are below the CAO of 550 µg/L west of Bairoko Street, but exceed the CAO in wells 54MW27, 54MW30, and 54MW32 located on the east side of Bairoko Street. Comparison of benzene distribution with screen placement (see Table 1) shows that benzene concentrations exceeding the CAO are confined to wells screened between approximately 15 and 25 feet below ground surface (bgs) and that benzene concentrations decrease to levels that are significantly below the CAO in wells screened deeper than 25 feet bgs (refer to Table 1 for wells 54MW29, 54MW31, 54MW33, 54MW35, 54MW37, and 510DW2).

Figure 3 also shows that the extent of benzene contamination between 15 and 25 feet bgs is not defined north of 54MW32.

TCE – 54MW34

Monitoring well 54MW34 was installed to determine the extent of TCE along the southern portion of SWMU 54 because low concentrations of TCE were detected in previously installed well 54MW18. TCE was not detected in well 54MW34 during the April 2010 sampling event, indicating that a separate TCE source does not exist.

Conclusions and Recommendations

The data collected in April 2010 indicate the horizontal extent of benzene in the shallow zone on the west side of Bairoko Street is defined to the CAO of 550 µg/L. However, an isolated area of elevated benzene contamination was detected in shallow zone monitoring well 54MW34 on the east side of Bairoko Street. Because 54MW34 is also separated by shallow zone wells 54MW02 and 54MW03 that have significantly lower benzene concentrations, it is likely that the benzene detected in 54MW34 is unrelated to the release that occurred on the west side of Bairoko Street.

The April 2010 data also show that within the deep zone, benzene concentrations exceeding the CAO are confined to the east side of Bairoko Street in wells screened between approximately 15 and 25 feet bgs. The data also show that benzene concentrations decrease to levels that are below the CAO at depths greater than 25 feet bgs.

Lastly, TCE was not detected in 54MW34, indicating that a separate TCE source does not exist.

Based on these findings, CH2M HILL recommends the following:

- **Shallow Zone (east side of Bairoko Street):** Install two monitoring wells at the locations shown on Figure 2 to determine the extent of benzene in groundwater south and east of well 54MW54. The new monitoring wells will be screened from 5 to 15 feet bgs and

groundwater will be analyzed for benzene using U.S. Environmental Protection Agency (EPA) Method 8260B.

- Deep Zone (east side of Bairoko Street): Install one monitoring well at the location shown on Figure 3 to define the extent of benzene in the deep zone beneath SWMU 54. The new monitoring well will be screened from 15 to 25 feet bgs and groundwater will be analyzed for benzene using EPA Method 8260B.
- Survey the locations and elevations of the newly installed wells.

References

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL). 2009a. *Pilot Study Work Plan for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. January.

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL). 2009b. *Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. October.

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL). 2010. *Phase II Additional Sampling Requirements for SWMU 54*. Prepared for Naval Facilities Engineering Command Southeast. February.

Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

TABLE 1
 Summary of Benzene Analytical Results
Naval Activity Puerto Rico, Ceiba, Puerto Rico

Well Identification	Screened Interval (ft bgs)	Sep-09	Dec-09	Jan-10	Apr-10
510DW1	19.6 – 24.6	--	--	21.8	--
510DW2	39.2 – 44.2	--	--	<5	--
510MW1	3.2 – 13.2	--	--	<5	--
510MW2	3.2 – 18.2	--	--	<5	--
510MW3	4.9 – 14.9	--	--	<5	--
510MW4	4.8 – 14.8	--	--	<5	--
54MW01	3.9 – 13.9	707 JB	--	653	--
54MW02	4.8 – 14.8	394 JB	--	500	--
54MW03	4.5 – 14.5	347 JB	--	357	--
54MW04	6.3 – 21.3	2.83 JB	--	<5	--
54MW05	6.3 – 21.3	2.2 JB	--	1.52 J	--
54MW06	5.0 – 15.0	9,260 JB	--	14,200	--
54MW19	15.3 – 25.3	NI	NI	45.2	--
54MW20	4.9 – 14.9	NI	NI	208	--
54MW21	15.5 – 25.5	NI	NI	15.2	--
54MW22	5.1 – 15.1	NI	NI	20.7	--
54MW23	15.2 – 25.2	NI	NI	<5	--
54MW24	5.2 – 15.2	NI	NI	<5	--
54MW25	15.5 – 25.5	NI	NI	<5	--
54MW26	4.9 – 14.9	NI	NI	<5	--
54MW27	15.2 – 25.2	NI	NI	7,410	--
54MW28	4.8 – 14.8	NI	NI	<5	--
54MW29	25.3 – 40.3	NI	NI	NI	2.45 J
54MW30	14.4 – 24.4	NI	NI	NI	621
54MW31	25.1 – 40.1	NI	NI	NI	31.4
54MW32	14.1 – 24.1	NI	NI	NI	1,100
54MW33	25.9 – 40.9	NI	NI	NI	238
54MW34	5.1 – 15.1	NI	NI	NI	10,800
54MW35	25.3 – 40.3	NI	NI	NI	6.17
54MW36	15.1 – 25.1	NI	NI	NI	330

TABLE 1

Summary of Benzene Analytical Results
Naval Activity Puerto Rico, Ceiba, Puerto Rico

Well Identification	Screened Interval (ft bgs)	Sep-09	Dec-09	Jan-10	Apr-10
54MW37	25.2 – 40.2	NI	NI	NI	13
54MW38	14.9 – 24.9	NI	NI	NI	117
54MW39	14.8 – 24.8	NI	NI	NI	<5
54MW40	4.8 – 14.8	NI	NI	NI	0.74 J
54MW41	15.0 – 25.0	NI	NI	NI	115

Notes:

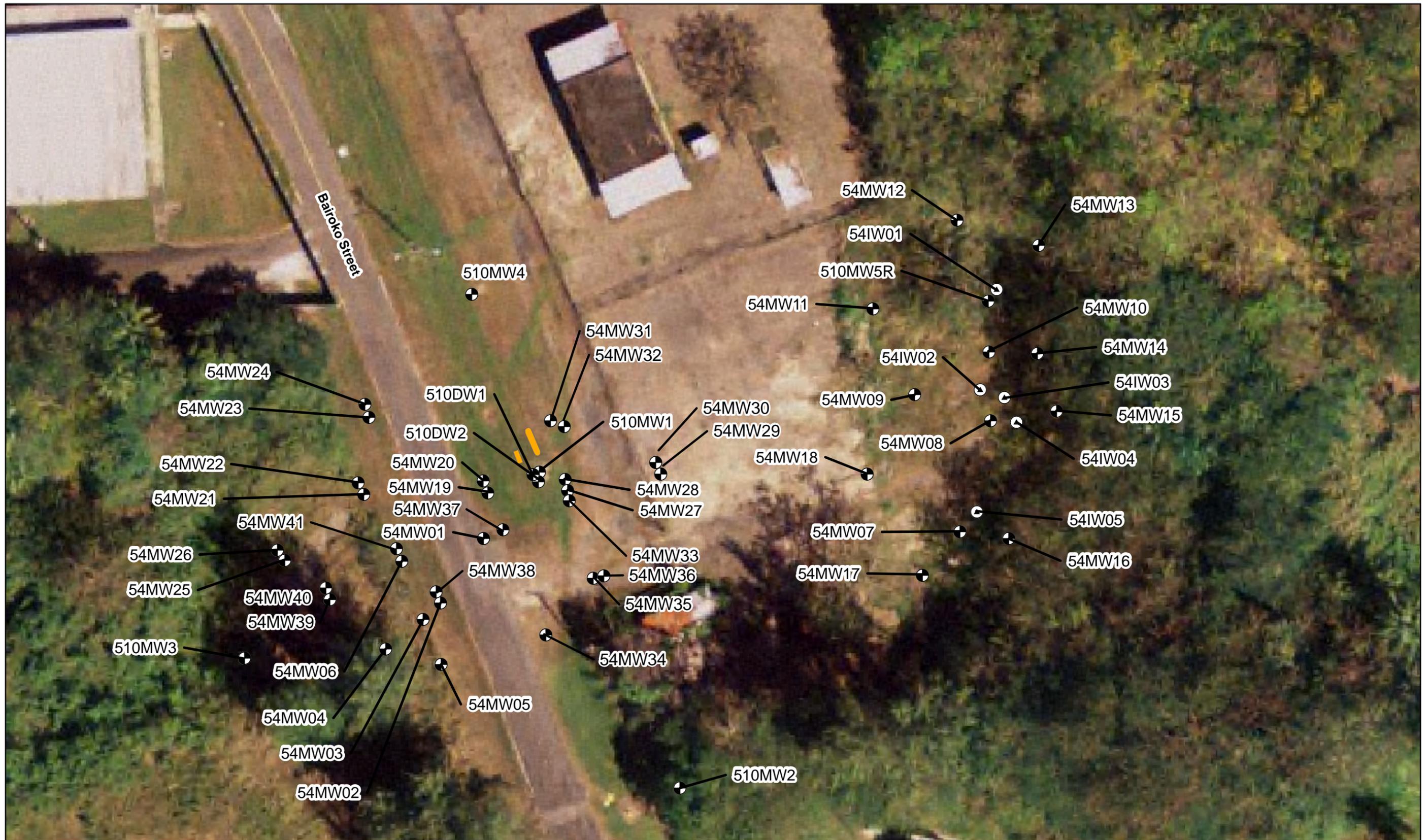
B = Indicates the analyte was detected in the associated method blank.

J = The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NI = the well was not yet installed

-- = the well was not sampled

Bold indicates the measured concentration exceeds the benzene CAO of 550 µg/L.



- Monitoring Well Location
- ▲ Proposed Injection Well Location
- Former Structure

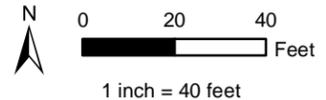
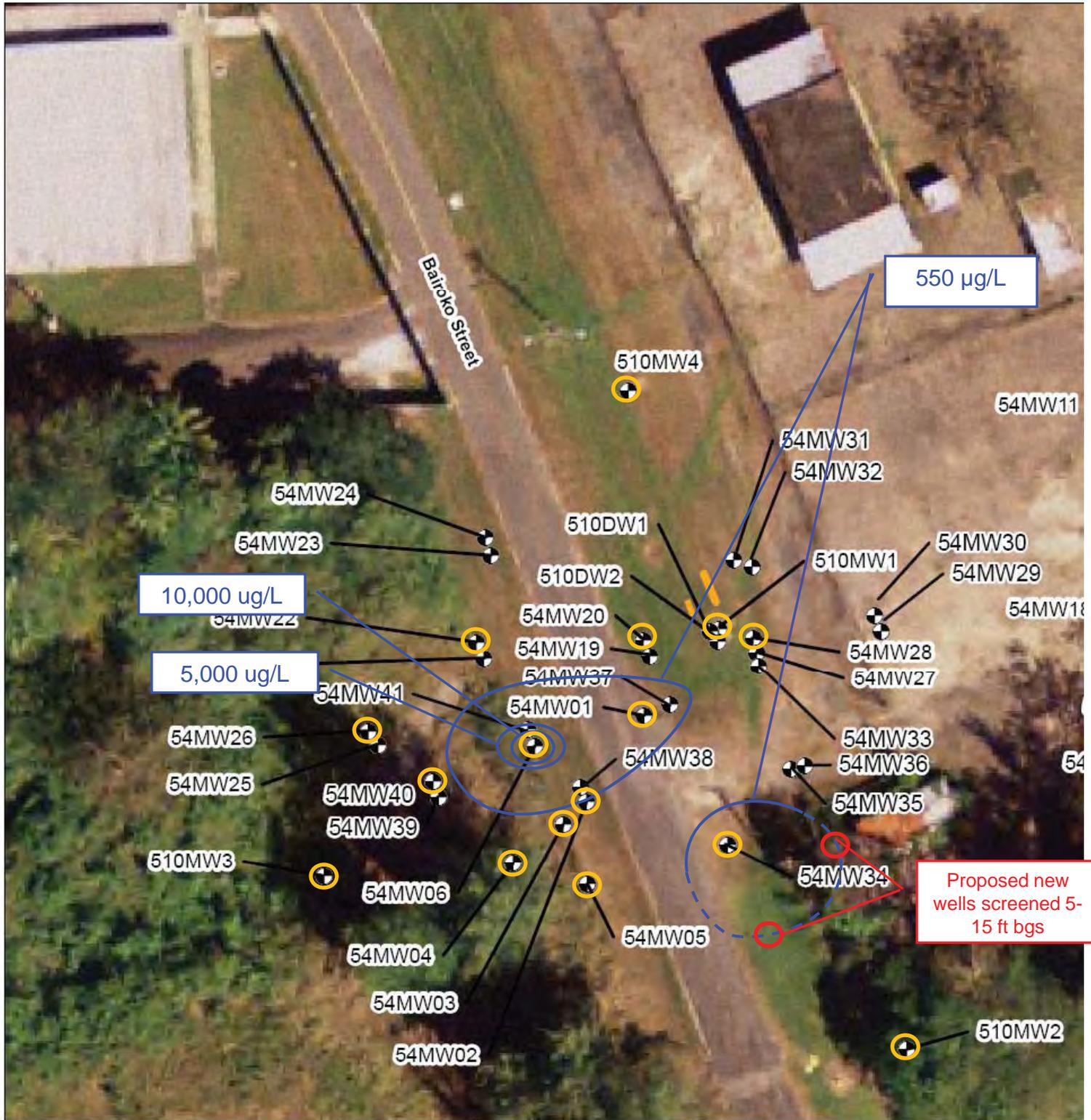


FIGURE 1
 Site Location Map
 SWMU 54
 Naval Activity, Puerto Rico

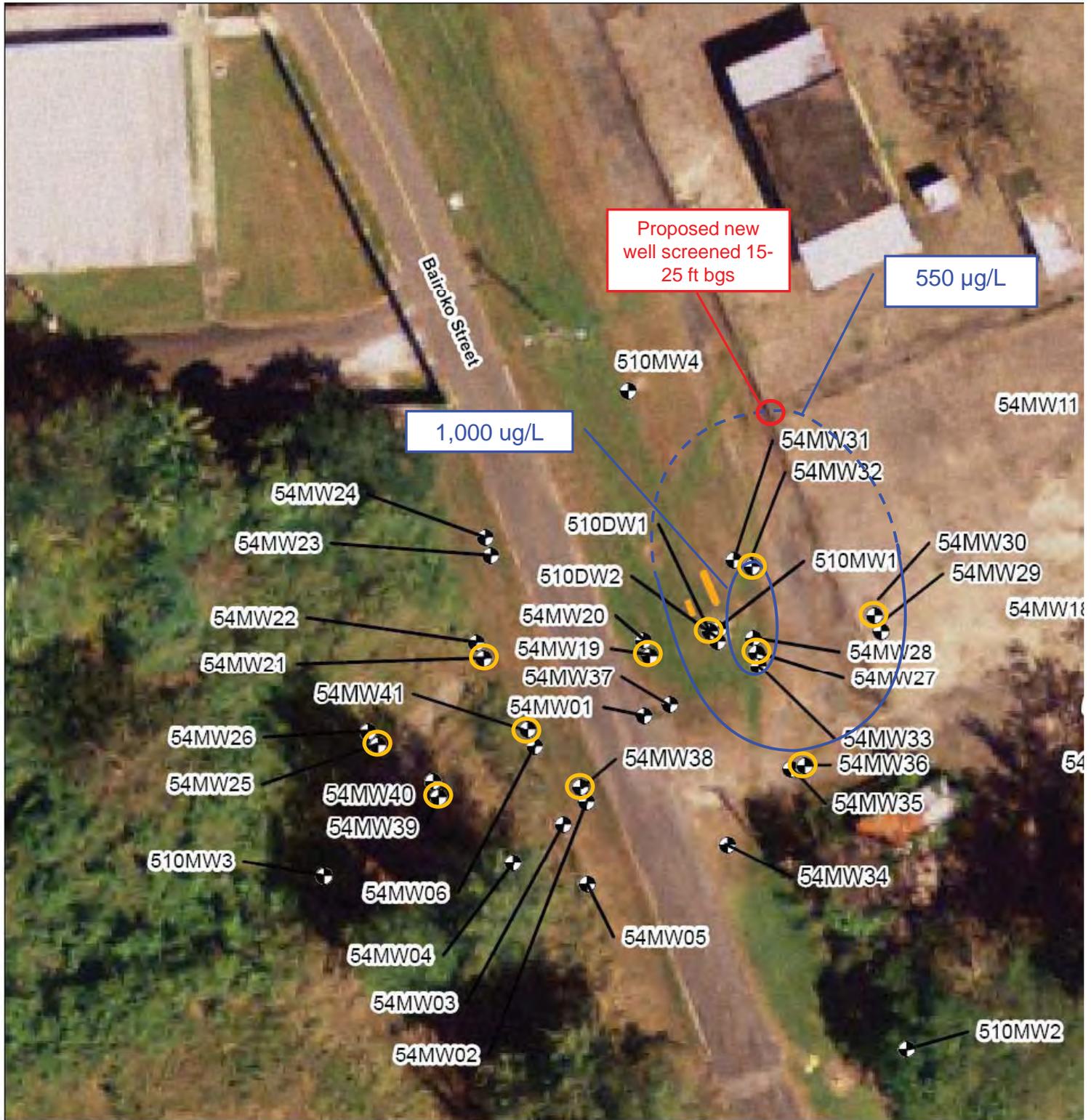


● Monitoring Well Location

● Monitoring Wells Screened Between 5 and 15 ft bgs

Note: Benzene corrective action objective (CAO) is 550 micrograms per liter ($\mu\text{g/L}$)

FIGURE 2
Distribution of Benzene in Groundwater - Shallow Zone
SWMU 54
Naval Activity Puerto Rico



● Monitoring Well Location

● Monitoring Wells Screened Between 15 and 25 ft bgs

Note: Benzene corrective action objective (CAO) is 550 micrograms per liter ($\mu\text{g/L}$)

FIGURE 3
 Distribution of Benzene in Groundwater - Deep Zone
 SWMU 54
 Naval Activity Puerto Rico



TECHNICAL MEMORANDUM

Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 55, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/NAVFAC SE
Tim Gordon/USEPA
Wilmarie Rivera/PR EQB

COPIES: David Criswell/NAVFAC SE
Pedro Ruiz/NAPR
Mark Kimes/Baker

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: September 15, 2009

AGVIQ-CH2M HILL has been retained by the Department of the Navy, Naval Facilities Engineering Command Southeast (NAVFAC SE) to conduct pilot testing at solid waste management unit (SWMU) 55 located at Naval Activity Puerto Rico (NAPR) formerly known as Naval Station Roosevelt Roads (NSRR), Ceiba, Puerto Rico. This work is performed under Contract Number N62470-08-D-1006, Contract Task Order (CTO) JM04. As detailed in the *Pilot Study Work Plan for SWMU 55* (CH2M HILL, 2009), the pilot testing will be conducted to evaluate the use of in situ chemical oxidation (ISCO) with potassium permanganate (KMnO₄) to remediate contaminated groundwater exceeding the corrective action objectives (CAOs) as described in the November 2005 Final Corrective Measures Study (CMS) Final Report (Baker Environmental, Inc. [Baker], 2005). According to the *Pilot Study Work Plan*, the ISCO pilot test at SWMU 55 will include a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, a total oxidant demand (TOD) study, ISCO injections, and quarterly monitoring. To date, the first phase of the pilot testing program, the baseline sampling event for existing wells, has been completed and installation of the pilot test injection wells is in progress. This technical memorandum (TM) is intended to document changes to the *Pilot Study Work Plan* and additional site characterization required in response to the baseline sampling event analytical results.

Baseline Sampling Event Results

The baseline sampling event at SWMU 55 was conducted in July 2009, during which, groundwater at 6 existing monitoring wells (7MW7, 7MW10, 7MW21, 7MW22, 7MW23, and 7MW24) was sampled and analyzed for TCE according to EPA Method SW846 8260B. The baseline sampling was conducted to verify results of the last sampling event (conducted

in 2003), evaluate the current extent of groundwater contamination, ensure the location of the TCE plume has not shifted since 2003, and possibly refine the pilot test injection well locations.

As summarized in Table 1, TCE was measured at monitoring well 7MW7 at 14,500 µg/L, an order of magnitude increase compared to the September 2003 measurement of 1,800 µg/L. In addition, there was also an order of magnitude increase in the TCE concentration in monitoring well 7MW23. The TCE concentration at 7MW24 remained essentially the same. The locations of the wells sampled and the estimated extent of TCE contamination in groundwater, using mining visualization software (MVS), are shown in Figure 1. The 2003 and 2009 analytical results are summarized in Table 1.

TABLE 1
 Summary of 2003 and 2009 TCE Analytical Results

Well Identification	Sample Date	TCE Concentration (µg/L)
7MW7	09/2003	1,800
	07/2009	14,500
7MW10	09/2003	<1
	07/2009	<5
7MW21	09/2003	<1
	07/2009	<5
7MW22	09/2003	<1
	07/2009	1.86
7MW23	09/2003	87
	07/2009	1,080
7MW24	09/2003	1,600
	07/2009	1,430

As shown in Figure 1, the extent of TCE in groundwater is not defined on the north, south, or east sides of the TCE plume. According to the *Final Corrective Measures Study Final Report for SWMUs 54 and 55* (Baker, 2005), TCE was measured (in 1999) at 1,500 µg/L at TWC, approximately 53 feet southeast of 7MW7 and (in 2003) at 66 µg/L at 7TCETW206, approximately 90 feet southeast of 7MW7 (Figure 2-12). Both of these locations could be upgradient of 7MW23 and 7MW24. In light of the significant increases in TCE concentrations at 7MW7 and 7MW23, and it is possible the TCE source area has not been adequately defined. In addition, the total downgradient extent (south of 7MW23) of TCE has not been delineated. Also, because the deepest existing monitoring wells are screened only about 25 feet below ground surface (ft bgs), the vertical extent of TCE contamination does not appear to be delineated.

Pilot Study Work Plan Changes

As a result of the baseline sampling event data, two changes were made to the pilot study work plan: an alteration in the preliminary injection test and the locations of the four ISCO injection wells.

The preliminary injection test was to be conducted to ensure it is possible to physically inject fluids in the site formation prior to delivering ISCO chemicals to the facility. Because of the high levels of TCE (> 14,000 µg/L) encountered in the pilot test injection area, it was determined the preliminary injection test was not warranted to avoid unintentional dispersion of the TCE plume without concurrent treatment. Also, rapid recharge of groundwater in monitoring wells was observed during groundwater sampling, indicating easy movement of groundwater in the subsurface. In the place of the preliminary injection test, falling and rising head slug tests will be completed to characterize the aquifer conductivity. It will be assumed that it will not be difficult to inject in an aquifer with significant conductivity.

In addition to replacing the preliminary injection test with a slug test, the locations of the ISCO injection wells were altered based on the baseline monitoring results. The new proposed injection well locations are shown on Figure 1. The locations and screen intervals of these wells were revised based on the baseline sampling results. The revised locations and screen intervals were selected to gain additional insight to the TCE concentrations in groundwater in the area of the 7MW07. The revised injection well locations and screen intervals are shown in Figure 1.

Recommended Path Forward

Based on the above information, CH2M HILL recommends a short delay of the SWMU 55 ISCO pilot test and installation of five monitoring well pairs to more fully delineate the TCE plume at SWMU 55. The proposed well pairs would be placed as shown in Figure 3 and consist of two screened intervals, 10-25 ft bgs and 25-40 ft bgs. These wells would be sampled for TCE and the data would then be considered part of the baseline sampling event. This data should improve the quality of the ISCO pilot test and our understanding of the plume by:

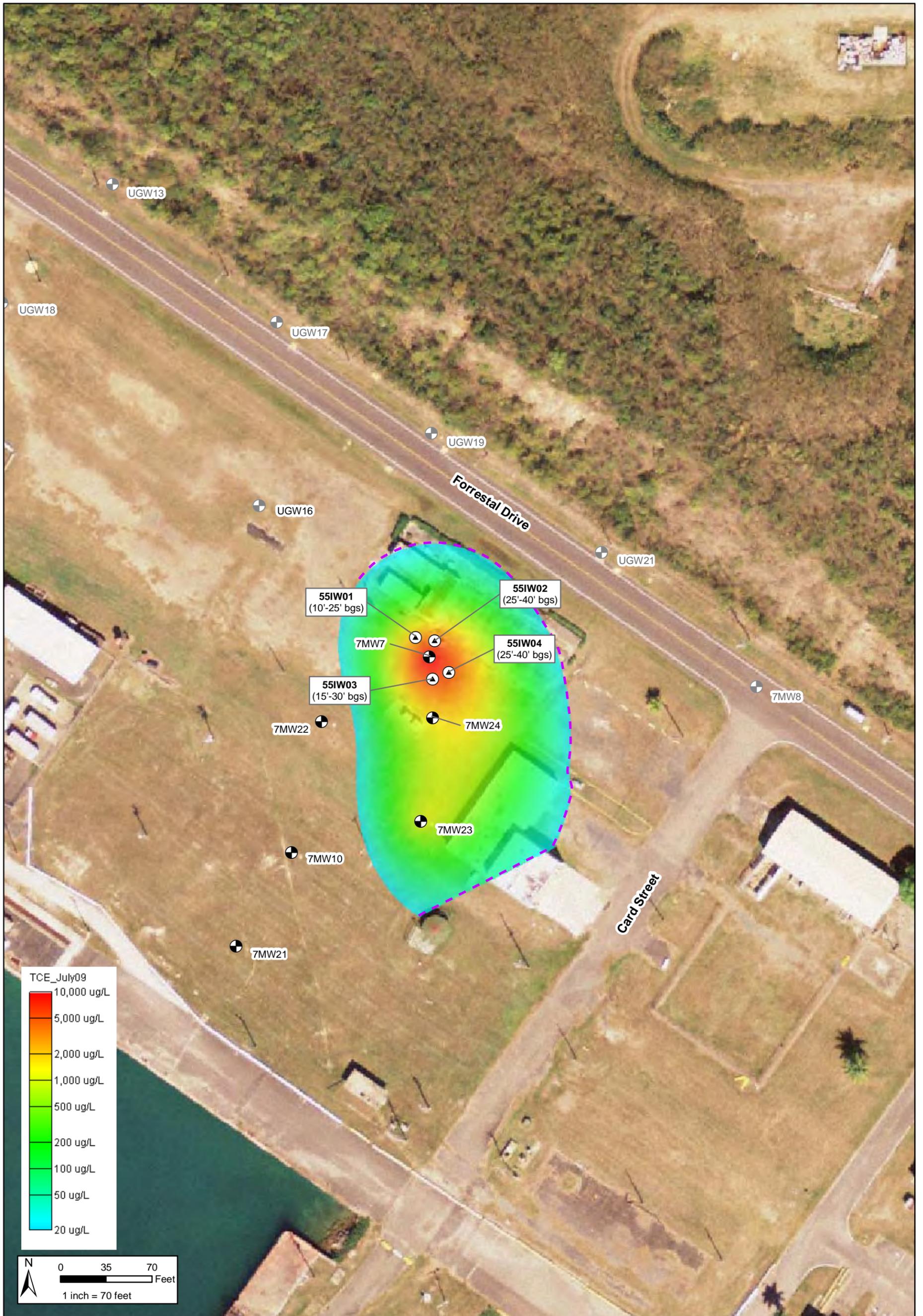
- Better defining the true source of TCE contamination and allowing us to focus the ISCO pilot test in the area of highest concentrations,
- Better defining the vertical extent of TCE in the source area so the ISCO targets the proper depth intervals, and;
- Delineating the downgradient extent of TCE contamination.

References

AGVIQ-CH2M HILL. 2009. *Pilot Study Work Plan for SWMU 55*. Prepared for Naval Facilities Engineering Command Southeast. January.

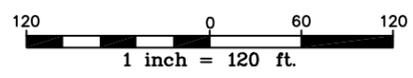
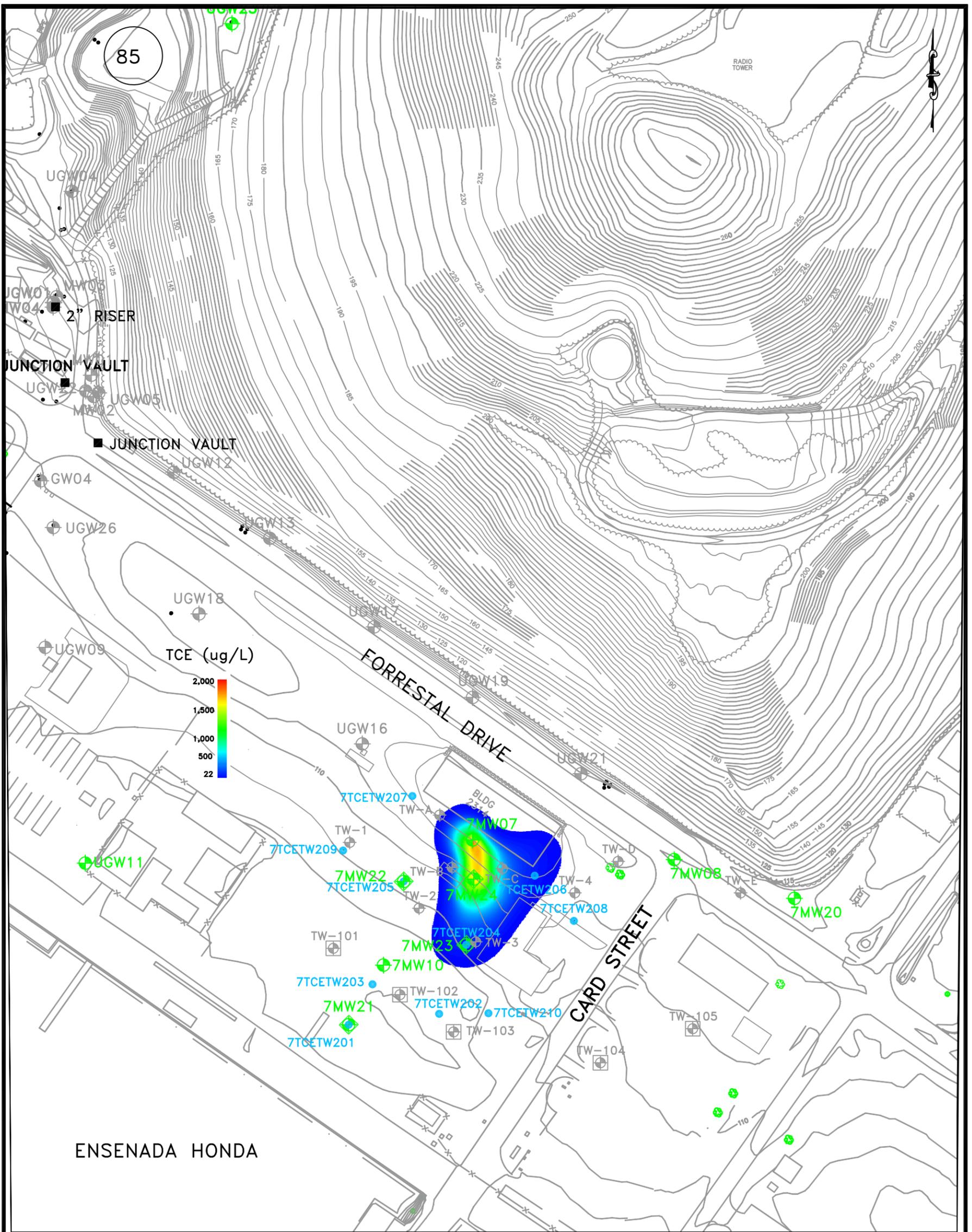
Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figures



- ⊕ Existing Monitoring Well Not Sampled in July 2009
- ⊙ Existing Monitoring Well Location
- ⊕ Proposed Injection Well Location
- - - Estimated Extent of TCE in Groundwater Exceeding 22 µg/L

FIGURE 1
 TCE Sampling Results - July 2009
 SWMU 55
 Naval Station Roosevelt Roads, Puerto Rico



NOTE:
 -DATUM PLAN USED IS MEAN LOW WATER = 100.00 FT. AS ESTABLISHED BY U.S. NAVY SURVEY SECTION AS OF NOVEMBER 1941.



k:\CH2M HILL II\CTO268 (100299)\DRAFT SWMU 54_55 CMS\CAD FIGURES\100299ch14

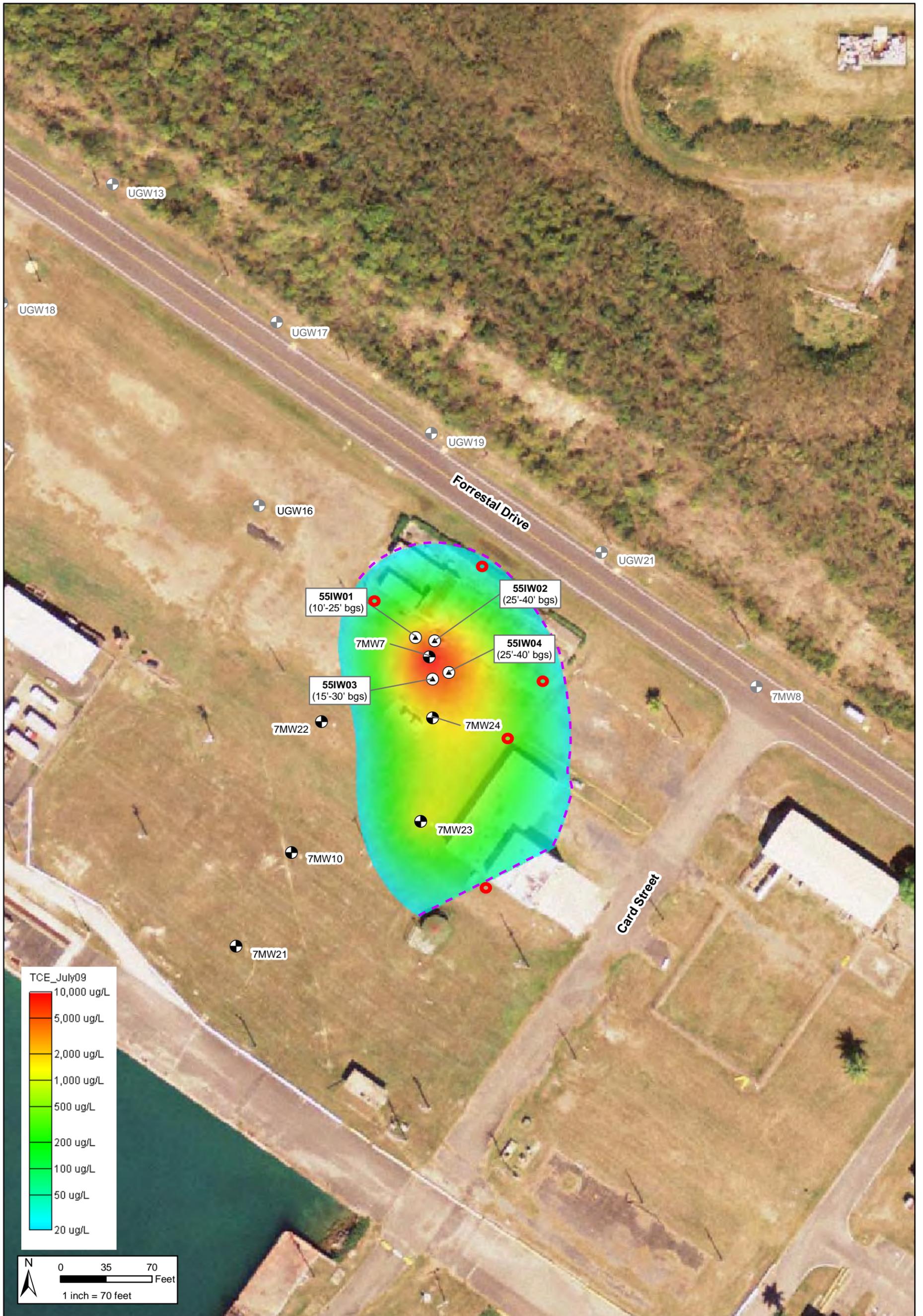
SOURCE: LANTDIV, FEB. 1992/1997

LEGEND

- ⊕ - EXISTING MONITOR WELL
- ⊕ - EXISTING MONITOR WELL - SAMPLED 2003
- - TEMPORARY WELL LOCATION - 2003
- ◆ - NEW MONITOR WELL LOCATION - 2003
- ⊕ - TEMPORARY MONITOR WELL LOCATION (SHALLOW AND DEEP) - 1999
- ⊕ - TEMPORARY MONITOR WELL LOCATION (DEEP ONLY) - 1999

FIGURE 2-12
TCE CONCENTRATIONS (ug/L), SEPTEMBER 2003
SWMU 55 - TCE PLUME NEAR TOW WAY FUEL FARM
CORRECTIVE MEASURES STUDY, OCTOBER 2004

NAVAL ACTIVITY PUERTO RICO
 PUERTO RICO



- ⊕ Existing Monitoring Well Not Sampled in July 2009
- ⊙ Existing Monitoring Well Location
- ⊕ Proposed Injection Well Location
- - - Estimated Extent of TCE in Groundwater Exceeding 22 µg/L

- Proposed Sample Well Pair Locations

Figure 3
 TCE Sampling Results - July 2009
 SWMU 55
 Naval Station Roosevelt Roads, Puerto Rico



TECHNICAL MEMORANDUM

Phase II Additional Sampling Requirements for SWMU 55, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/PMO SE
Pedro Ruiz/NAPR

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: December 23, 2009

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Base Closure and Realignment (BRAC) Program Management Office Southeast (PMO SE) to conduct pilot testing at solid waste management unit (SWMU) 55 located at Naval Activity Puerto Rico (NAPR) formerly known as Naval Station Roosevelt Roads (NSRR), Ceiba, Puerto Rico. This work is performed under Contract No. N62470-08-D-1006, Task Order JM04. As detailed in the *Pilot Study Work Plan for SWMU 55* (AGVIQ-CH2M HILL, 2009a), the pilot testing is in progress to evaluate the use of in situ chemical oxidation (ISCO) with permanganate to remediate contaminated groundwater with trichloroethene (TCE) levels exceeding the corrective action objectives (CAOs) described in the November 2005 *Final Corrective Measures Study Final Report* (Baker Environmental, Inc. [Baker], 2005). The ISCO pilot test at SWMU 55 includes a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, a total oxidant demand (TOD) study, ISCO injections, and quarterly monitoring (AGVIQ-CH2M HILL, 2009a). To date, the baseline sampling event for existing wells, installation of injection wells, and TOD testing have been completed. The ISCO injections are in progress.

This technical memorandum documents additional site characterization needed in response to the baseline sampling event, ISCO injection well sampling, and Phase I additional characterization sampling analytical results.

Baseline Sampling Event Results

The baseline sampling event at SWMU 55 was conducted in July 2009. During this event, groundwater from six existing monitoring wells (7MW7, 7MW10, 7MW21, 7MW22, 7MW23, and 7MW24) was sampled and analyzed for TCE according to U.S. Environmental Protection Agency (EPA) Method SW846 8260B. The baseline sampling was conducted to verify results of the previous sampling event (conducted in September 2003), evaluate the current extent of groundwater contamination, confirm that the location of the TCE plume has not shifted since 2003, and possibly refine the pilot test injection well locations.

As summarized in Table 1, three wells had TCE detections exceeding the CAO of 22 micrograms per liter ($\mu\text{g/L}$). TCE was measured at monitoring well 7MW7 at 14,500 micrograms per liter ($\mu\text{g/L}$), an order of magnitude increase compared to the September 2003 measurement of 1,800 $\mu\text{g/L}$. In addition, there was also an order of magnitude increase in the TCE concentration in monitoring well 7MW23. The TCE concentration at 7MW24 remained essentially the same. The locations of the wells sampled and the estimated extent of TCE contamination in groundwater, using mining visualization software (MVS), are shown on Figure 1. The 2003 and 2009 analytical results are summarized in Table 1.

TABLE 1
 Summary of 2003 and 2009 TCE Analytical Results
 Naval Activity Puerto Rico, Ceiba, Puerto Rico

Well Identification	Sample Date	TCE Concentration ($\mu\text{g/L}$)
7MW7	09/2003	1,800
	07/2009	14,500
7MW10	09/2003	<1
	07/2009	<5
7MW21	09/2003	<1
	07/2009	<5
7MW22	09/2003	<1
	07/2009	1.86
7MW23	09/2003	87
	07/2009	1,080
7MW24	09/2003	1,600
	07/2009	1,430

Notes:

Bold values indicate concentrations in excess of the CAO of 22 $\mu\text{g/L}$.

Based on the above data, the extent of TCE in groundwater was not defined on the north, south, or east sides of the TCE plume, the source area was not adequately defined, and the vertical extent of TCE contamination was not delineated (AGVIQ-CH2M HILL, 2009b).

Injection Well and Phase I Additional Characterization Sampling Event Results

Four ISCO injection wells (55IW01 through 55IW04) were installed in September 2009, and groundwater from these wells was sampled and analyzed for TCE according to EPA Method SW846 8260B, as directed in the *Pilot Study Work Plan for SWMU 55*. As summarized in Table 2, TCE was measured at concentrations between 137 and 33,700 $\mu\text{g/L}$.

Based on the results of the baseline sampling at existing wells, AGVIQ-CH2M HILL recommended installation and sampling of 10 additional wells to fully define the horizontal and vertical extent of TCE plume at SWMU 55 (AGVIQ-CH2M HILL, 2009b). These wells

(55MW01 through 55MW10) were installed during the same phase as the injection wells in September 2009. The Phase I additional baseline sampling event at SWMU 55 was conducted in November 2009. During this event, groundwater from the 10 new monitoring wells and 4 new injection wells was sampled and analyzed for TCE according to EPA Method SW846 8260B.

As summarized in Table 2, TCE was measured above the CAO of 22 µg/L at monitoring wells on the eastern and southeastern sides of the plume. The locations of injection and monitoring wells sampled and the estimated extent of TCE contamination in groundwater, using MVS, are shown on Figure 1.

TABLE 2
 Summary of November 2009 Phase I Additional Baseline TCE
 Analytical Results
Naval Activity Puerto Rico, Ceiba, Puerto Rico

Well Identification	Screen Interval (ft bgs)	TCE Concentration (µg/L)
55MW01	25-40	640
55MW02	10-25	71.2
55MW03	25-40	108
55MW04	10-25	7.6
55MW05	25-40	0.21 J
55MW06	10-25	7.2
55MW07	25-40	2.0 J
55MW08	10-25	3.1 J
55MW09	25-40	384
55MW10	10-25	184
55IW01	10-25	33,600
55IW02	25-40	1,660
55IW03	15-30	3,600
55IW04	25-40	137

Notes:
Bold values indicate concentrations in excess of the CAO of 22 µg/L.
 ft bgs = foot below ground surface

As shown on Figure 1, the horizontal extent of TCE in groundwater is still not defined on the east or southeast sides of the TCE plume. Also, the significant TCE detections in the injection wells screened to 40 ft bgs indicate that the vertical extent of TCE contamination in the source zone is not delineated.

Recommended Path Forward

Based on the above information, AGVIQ-CH2M HILL recommends the installation of 10 new monitoring wells to more fully delineate the horizontal and vertical extent of the

TCE plume at SWMU 55. The proposed wells (55MW11 through 55MW20) would be placed as shown on Figure 1 and screened according to the intervals indicated on Figure 1. AGVIQ-CH2M HILL recommends sampling groundwater from these wells for TCE and considering the data as part of the baseline sampling event. These data should improve the definition and characterization of the TCE plume in the following ways:

- Better defining the true source of TCE contamination and allowing potential future ISCO injections to be focused in the appropriate areas
- Better defining the vertical extent of TCE in the source area so that the ISCO targets the proper depth intervals
- Delineating the downgradient extent of TCE contamination

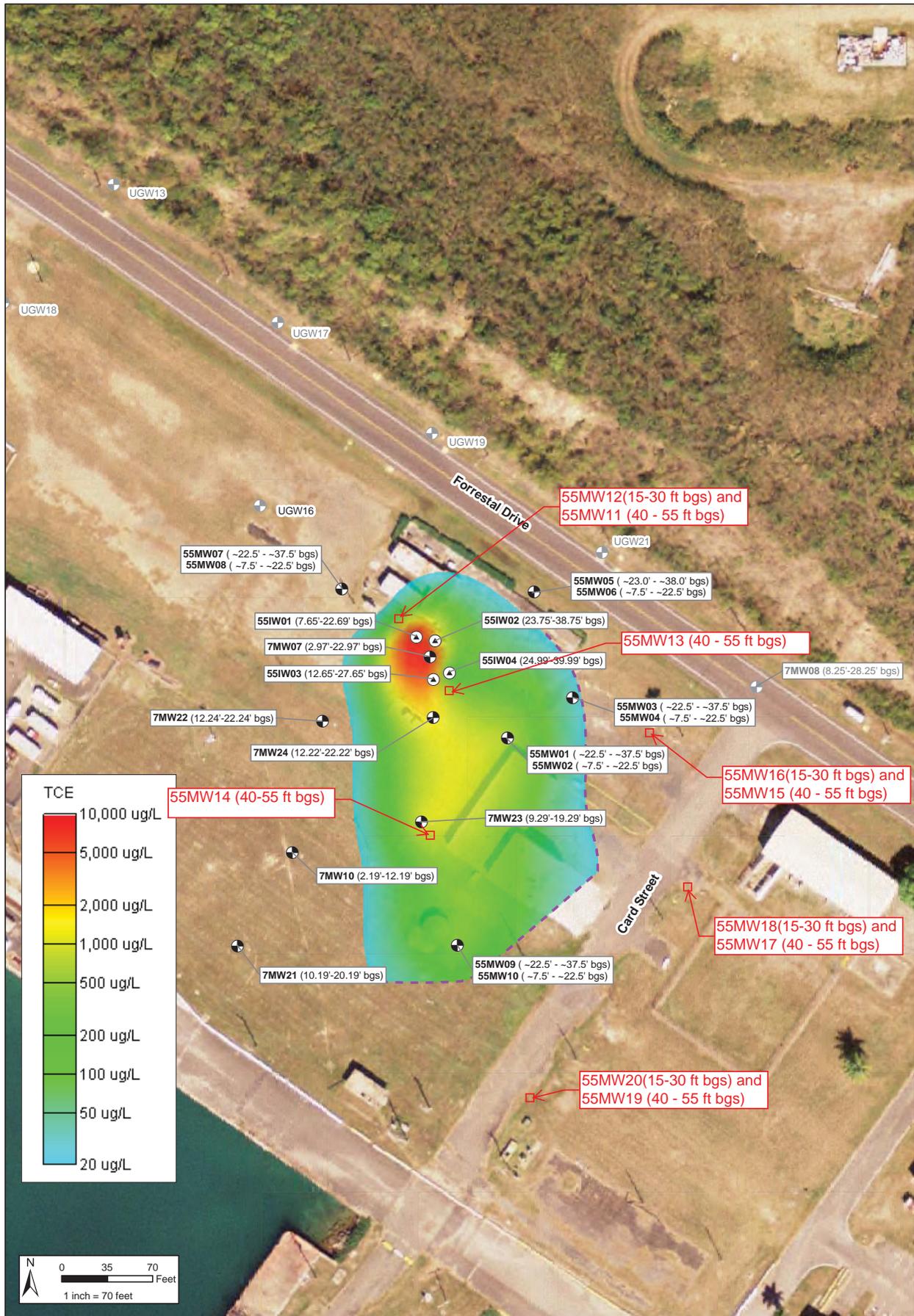
References

AGVIQ-CH2M HILL. 2009a. *Pilot Study Work Plan for SWMU 55*. Prepared for Naval Facilities Engineering Command Southeast. January.

AGVIQ-CH2M HILL. 2009b. *Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 55 Technical Memorandum*.

Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figure



- ⊕ Existing Monitoring Well Not Included in Baseline Sampling Events
- ⊙ Existing Monitoring Well Location
- ⊕ Injection Well Location
- - - Estimated Extent of TCE in Groundwater Exceeding 22 µg/L

FIGURE 1
TCE Sampling Results - November 2009
SWMU 55
Naval Station Roosevelt Roads, Puerto Rico



TECHNICAL MEMORANDUM

Phase III Additional Sampling Requirements for SWMU 55, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/PMO SE
Pedro Ruiz/NAPR

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: March 4, 2010

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Base Closure and Realignment (BRAC) Program Management Office Southeast (PMO SE) to conduct a pilot-scale study at Solid Waste Management Unit (SWMU) 55 located at Naval Activity Puerto Rico (NAPR), formerly known as Naval Station Roosevelt Roads (NSRR), Ceiba, Puerto Rico. This work is being performed under Contract No. N62470-08-D-1006, Task Order JM04. As detailed in the *Pilot Study Work Plan for SWMU 55* (AGVIQ-CH2M HILL, 2009a), the pilot testing is in progress to evaluate the use of in situ chemical oxidation (ISCO) with permanganate to remediate contaminated groundwater with trichloroethene (TCE) levels exceeding the corrective action objectives (CAOs) described in the November 2005 *Final Corrective Measures Study Final Report* (Baker Environmental, Inc., 2005). The ISCO pilot test at SWMU 55 includes a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, a total oxidant demand (TOD) study, ISCO injections, and quarterly monitoring (AGVIQ-CH2M HILL, 2009a). To date, the baseline sampling event for existing wells, installation of injection wells and additional monitoring wells, TOD testing, the ISCO injections, and the first quarterly monitoring event have been completed.

This technical memorandum documents additional site characterization needed in response to the baseline, ISCO injection well, and Phase I and Phase II additional characterization sampling events.

Baseline Sampling Event Results

The baseline sampling event at SWMU 55 was conducted in July 2009. During this event, groundwater from six existing monitoring wells (7MW7, 7MW10, 7MW21, 7MW22, 7MW23, and 7MW24) was sampled and analyzed for TCE according to U.S. Environmental Protection Agency (EPA) Method SW846 8260B. The baseline sampling was conducted to verify results of the previous sampling event (conducted in September 2003), evaluate the current extent of groundwater contamination, confirm that the location of the TCE plume has not shifted since 2003, and possibly refine the pilot test injection well locations.

As summarized in Table 1, three wells had TCE detections exceeding the CAO of 22 micrograms per liter ($\mu\text{g/L}$). TCE was measured at monitoring well 7MW7 at 14,500 $\mu\text{g/L}$, an order of magnitude increase compared to the September 2003 measurement of 1,800 $\mu\text{g/L}$. In addition, there was also an order of magnitude increase in the TCE concentration in monitoring well 7MW23. The TCE concentration at 7MW24 remained essentially the same. The locations of the wells sampled and the estimated extent of TCE contamination in groundwater are shown on Figure 1. As illustrated, the extent of TCE in groundwater was not defined on the north, south, or east sides of the TCE plume during the baseline event, and the source area was not adequately defined. Additionally Figure 2 illustrates that the vertical extent of TCE contamination has not been delineated (AGVIQ-CH2M HILL, 2009b).

TABLE 1
 Summary of TCE Analytical Results
Naval Activity Puerto Rico, Ceiba, Puerto Rico

Well ID	Screened Interval	TCE Concentration ($\mu\text{g/L}$)			
		Sep-03	Jul-09	Nov-09	Feb-10
7MW07	Shallow	1,800	14,500	--	--
7MW10	Shallow	<1	<5	--	--
7MW21	Shallow	<1	<5	--	--
7MW22	Shallow	<1	1.86	--	--
7MW23	Shallow	87	1,080	--	--
7MW24		1,600	1,430	--	--
55IW01	Deep	--	--	33,600	--
55IW02	Shallow	--	--	1,660	--
55IW03	Deep	--	--	3,600	--
55IW04	Shallow	--	--	137	--
55MW01	Deep	--	--	640	--
55MW02	Shallow	--	--	71	--
55MW03	Deep	--	--	108	--
55MW04	Shallow	--	--	7.6	--
55MW05	Deep	--	--	0.2J	--
55MW06	Shallow	--	--	7.2	--
55MW07	Deep	--	--	2.0J	--
55MW08	Shallow	--	--	3.1J	--
55MW09	Deep	--	--	384	--
55MW10	Shallow	--	--	184	--
55MW11	Deep	--	--	--	243

TABLE 1
 Summary of TCE Analytical Results
Naval Activity Puerto Rico, Ceiba, Puerto Rico

55MW12	Shallow	--	--	--	13.1
55MW13		Not Installed			
55MW14	Deep	--	--	--	1,090
55MW15	Deep	--	--	--	46
55MW16	Shallow	--	--	--	5.46
55MW17	Shallow	--	--	--	177
55MW18	Deep	--	--	--	2.0J
55MW19	Deep	--	--	--	1.7J
55MW20	Shallow	--	--	--	0.7J

Notes:

Bold values indicate concentrations in excess of the CAO of 22 µg/L.

J = value was measured below its practical quantitation limit.

Injection Well and Phase I Additional Characterization Sampling Event Results

Four ISCO injection wells (55IW01 through 55IW04) (see Figures 1 and 2) were installed in September 2009, and groundwater from these wells was sampled and analyzed for TCE according to EPA Method SW846 8260B, as directed in the *Pilot Study Work Plan for SWMU 55*. As summarized in Table 1, TCE was measured at concentrations between 137 and 33,600 µg/L.

Based on the results of the baseline sampling at existing wells, AGVIQ-CH2M HILL recommended installation and sampling of 10 additional wells (55MW01 through 55MW10) (see Figures 1 and 2) to fully define the horizontal and vertical extent of TCE plume at SWMU 55 (AGVIQ-CH2M HILL, 2009b). These wells were also installed in September 2009. The Phase I additional baseline sampling event at SWMU 55 was conducted in November 2009. During this event, groundwater from the 10 new monitoring wells (55MW01 through 55MW10) and four new injection wells (55IW01 through 55IW04) was sampled and analyzed for TCE according to EPA Method SW846 8260B.

As summarized in Table 1, TCE was detected above the CAO of 22 µg/L at monitoring wells located on the eastern and southeastern sides of the plume (55MW01, 55MW02, 55MW03, 55MW09, and 55MW10). The locations of injection and monitoring wells sampled are shown on Figures 1 and 2.

Phase II Additional Characterization Sampling Event Result

In January and February 2010, 10 additional monitoring wells (55MW11 through 55MW20) were installed to complete the horizontal and vertical delineation of TCE contamination at SWMU 55 (AGVIQ-CH2M HILL, 2009c), and groundwater from these wells was sampled and analyzed for TCE according to EPA Method SW846 8260B. As summarized in Table 1, TCE was measured at concentrations between 0.7 and 1,090 µg/L. TCE was measured above

the CAO of 22 µg/L at monitoring wells on the eastern and western sides of the plume (55MW 11 and 55MW17). The locations of monitoring wells sampled and the estimated extent of TCE contamination in groundwater (using all TCE data collected since July 2009) are shown on Figures 1 and 2.

As shown on Figures 1 and 2, the horizontal extent of TCE in groundwater is still not defined on the east or west sides of the TCE plume. Also, the significant TCE detection in monitoring well 55MW14 (screened to 40 feet below ground surface) indicates that the vertical extent of TCE contamination in the source area has not been delineated.

Recommended Path Forward

Based on the above information, AGVIQ-CH2M HILL recommends the installation of four new monitoring wells to delineate the horizontal and vertical extent of the TCE contamination beneath SWMU 55. The proposed wells (55MW21 through 55MW24) would be installed and screened according to the intervals shown on Figure 3.

AGVIQ-CH2M HILL recommends sampling groundwater from these wells for TCE and considering the data as part of the baseline sampling event. These data should improve the definition and characterization of the TCE plume in the following ways:

- Better defining the vertical extent of TCE in the source area so that the ISCO targets the proper depth intervals
- Delineating the downgradient extent of TCE contamination to confirm that direct discharge to surface water is not occurring

References

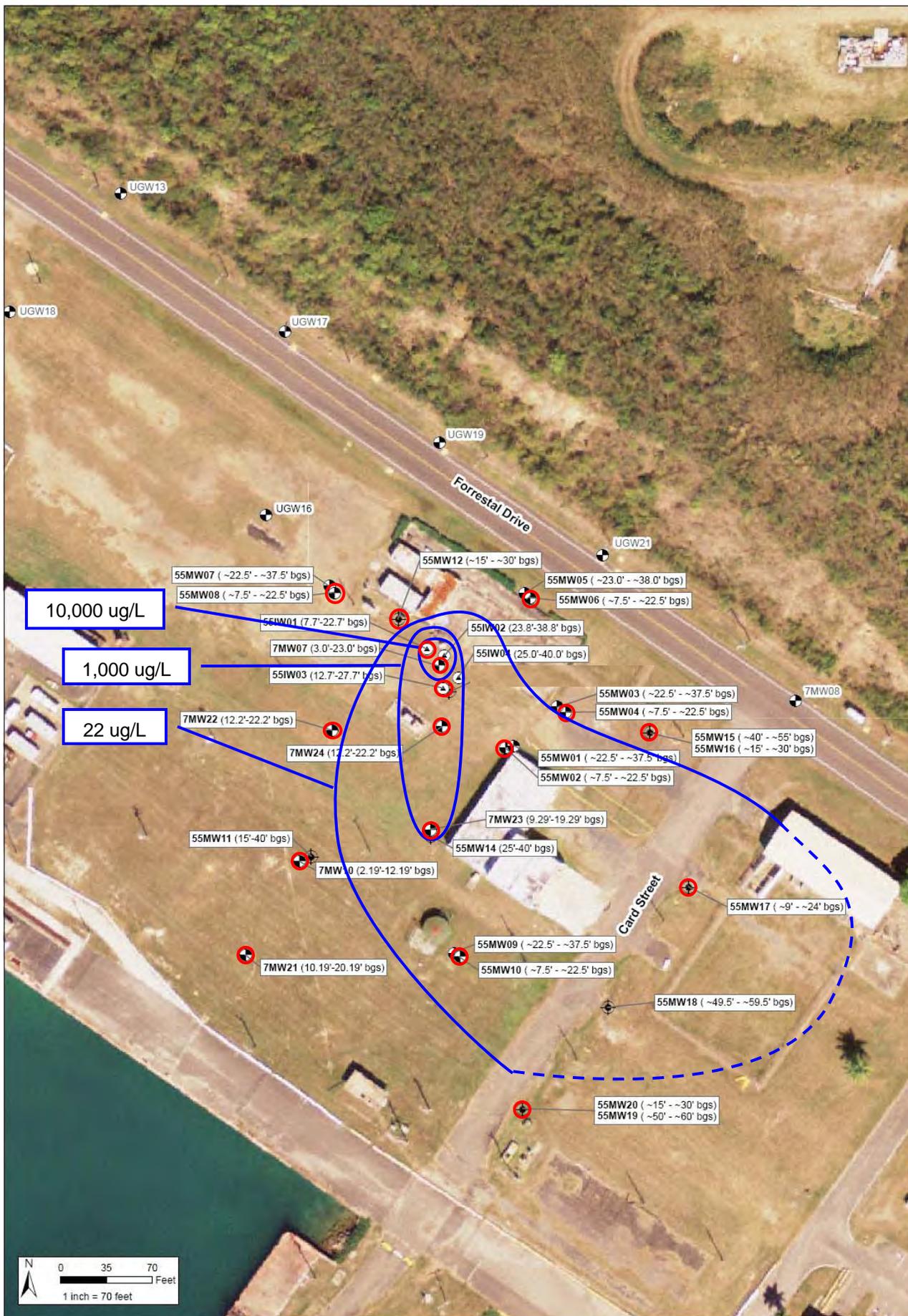
AGVIQ-CH2M HILL. 2009a. *Pilot Study Work Plan for SWMU 55*. Prepared for Naval Facilities Engineering Command Southeast. January.

AGVIQ-CH2M HILL. 2009b. *Revised Pilot Study Injection Well Locations and Additional Sampling Requirements for SWMU 55 Technical Memorandum*.

AGVIQ-CH2M HILL. 2009c. *Phase II Additional Sampling Requirements for SWMU 55 Technical Memorandum*. December.

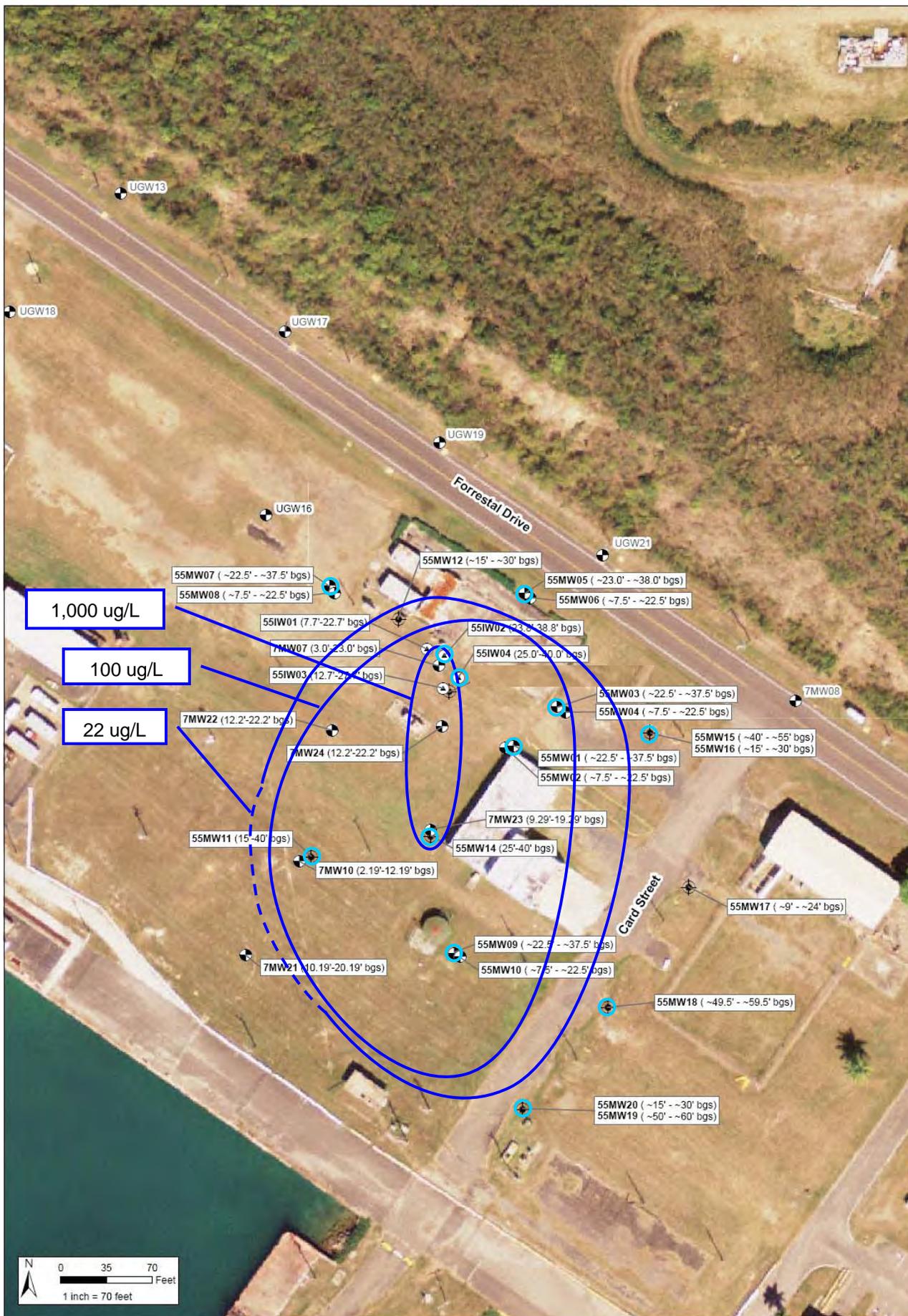
Baker Environmental, Inc. 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figures



- Existing Monitoring Well Not Included in Baseline Sampling Events
- Existing Monitoring Well Location
- ⊕ Injection Well Location
- ⊕ Monitoring Well Not Surveyed
- Wells screened primarily less than 25 ft bgs used to create isocontours.

FIGURE 1
 TCE Sampling Results – Shallow Zone (0-25 ft bgs)
 SWMU 55
 Naval Station Roosevelt Roads, Puerto Rico



- Existing Monitoring Well Not Included in Baseline Sampling Events
- Existing Monitoring Well Location
- Injection Well Location
- Monitoring Well Not Surveyed
- Wells screened primarily greater than 25 ft bgs used to create isocontours.

FIGURE 2
 TCE Sampling Results – Deep Zone (>25 ft bgs)
 SWMU 55
 Naval Station Roosevelt Roads, Puerto Rico



FIGURE 3
Proposed Well Locations
SWMU 55
Naval Station Roosevelt Roads, Puerto Rico



TECHNICAL MEMORANDUM

Phase IV Additional Sampling Requirements and Revised Technical Approach for SWMU 55, Naval Activity Puerto Rico, Ceiba, Puerto Rico

PREPARED FOR: Mark Davidson/PMO SE
Pedro Ruiz/NAPR

PREPARED BY: Amanda Struse/CH2M HILL
Doug Downey/CH2M HILL
Tom Beisel/CH2M HILL

DATE: June 26, 2010

AGVIQ-CH2M HILL Joint Venture III (AGVIQ-CH2M HILL) has been retained by the Department of the Navy, Base Closure and Realignment (BRAC) Program Management Office Southeast (PMO SE) to conduct a pilot study at Solid Waste Management Unit (SWMU) 55 located at Naval Activity Puerto Rico (NAPR), formerly known as Naval Station Roosevelt Roads (NSRR), Ceiba, Puerto Rico. This work is being performed under Contract No. N62470-08-D-1006, Task Order JM04. As detailed in the *Pilot Study Work Plan for SWMU 55* (AGVIQ-CH2M HILL, 2009), the pilot study is in progress to evaluate the use of in situ chemical oxidation (ISCO) with permanganate to remediate contaminated groundwater with trichloroethene (TCE) levels exceeding the corrective action objectives (CAOs) described in the November 2005 *Final Corrective Measures Study Final Report* (Baker Environmental, Inc. [Baker], 2005). The ISCO pilot study at SWMU 55 includes a baseline site characterization sampling event, a preliminary injection test, installation of injection wells, a total oxidant demand (TOD) study, ISCO injections, and quarterly monitoring (AGVIQ-CH2M HILL, 2009). To date, the baseline sampling event for existing wells, installation of injection wells, TOD testing, one ISCO injection, and the first and second quarterly monitoring events have been completed. In addition, several groundwater monitoring wells have been installed to complete characterization of TCE concentrations in groundwater.

This technical memorandum summarizes the current conceptual site model (CSM), including the additional site characterization conducted in response to the groundwater sampling results, the results of the ISCO injection, and recommendation for the future technical approach for groundwater remediation at the site.

CSM Summary

In response to data collected during the baseline sampling event in July 2009, additional characterization of TCE contamination in groundwater was conducted at SWMU 55 to fully delineate the extent of TCE contamination in groundwater both laterally and vertically. To date, a total of 23 monitoring wells (55MW01 through 55MW23) and four injection wells

(55IW01 through 55IW04) have been installed and sampled for TCE (Figure 1). Generally, these wells were installed with 15-foot screens and depths varying between 24 and 68 feet below ground surface (bgs). Many of these wells, combined with the existing monitoring wells 7MW10, 7MW21, 7MW22, 7MW23, and 7MW24, also comprised the monitoring network for the ISCO injection, as discussed later in the text.

Lithologic samples were collected during installation of all new monitoring wells, and the geologic observations correspond well with the geology described in the *Final Corrective Measures Study Final Report* (Baker, 2005). The hydrologic units (weathered rock and partially weathered rock) and associated aquifer characterizations, such as general depth to groundwater, hydraulic gradient, groundwater velocity, and hydraulic conductivity, are also consistent with the previous report, with the exception of the extent of TCE contamination present in groundwater at SWMU 55. There appears to be a single hydrologic unit with no significant upward or downward gradients.

Groundwater data acquired during the baseline sampling event at the existing monitoring wells and Phase I through Phase III Additional Characterization events were used collectively to establish the baseline TCE distribution in groundwater at SWMU 55. The analytical results are summarized in Table 1 and Figures 2 through 4. Groundwater analytical results were interpreted using Mining Visualization Software. To optimize the treatment area for ISCO injections, the groundwater results were divided into two vertical zones extending between approximately 15 and 25 feet bgs and 25 and 40+ feet bgs. Results are shown for groundwater at approximately 14 feet bgs (Figure 2), 25 feet bgs (Figure 3), and 41 feet bgs (Figure 4).

The greatest TCE concentrations were detected in the most shallow interval at injection well 55IW01 (33,600 micrograms per liter [$\mu\text{g/L}$]), defining the southern portion of the source area. A zone exceeding 1,000 $\mu\text{g/L}$ extends from the source area to the well pair 7MW23/55MW14. The 1,000 $\mu\text{g/L}$ area, including the source area, has been defined as the target treatment zone for SWMU 55. As shown on Figures 3 and 4, the TCE concentrations decline with groundwater elevation, however the lateral extent of TCE exceeding the CAO of 22 $\mu\text{g/L}$ increases slightly with depth.

TABLE 1
 Summary of TCE Analytical Results
 Naval Activity Puerto Rico

Well ID	Screened Interval (feet bgs)	TCE Concentration ($\mu\text{g/L}$)			
		July 2009 (Baseline)	November 2009 (Baseline - Phase I)	January/February 2010 (1st Quarter & Baseline Phase II)	April 2010 (2nd Quarter & Baseline Phase III)
7MW07	10-25	14,500	--	0 ^a	252
7MW10	2-12	<5	--	1.21J	<5
7MW21	10-20	<5	--	<5	<5
7MW22	12-22	1.86	--	1.98J	2.02J
7MW23	9-19	1,080	--	1,330	990

TABLE 1
 Summary of TCE Analytical Results
 Naval Activity Puerto Rico

TCE Concentration (µg/L)					
Well ID	Screened Interval (feet bgs)	July 2009 (Baseline)	November 2009 (Baseline - Phase I)	January/February 2010 (1st Quarter & Baseline Phase II)	April 2010 (2nd Quarter & Baseline Phase III)
7MW24	12-22	1,430	--	0 ^a	14.4
55IW01	10.5-25.5	NI	33,600	0 ^a	3,750
55IW02	25-40	NI	1,660	0 ^a	694
55IW03	15.5-30.5	NI	3,600	0 ^a	27.2
55IW04	25-40	NI	137	0 ^a	38.8
55MW01	24.5-39.5	NI	640	648	787
55MW02	9-24	NI	71	1.16J	<5
55MW03	24-39	NI	108	98	64
55MW04	10-25	NI	7.6	6.09	7.62
55MW05	25.5-40.5	NI	0.2J	<5	<5
55MW06	10-25	NI	7.2	1.07J	<5
55MW07	25-40	NI	2.0J	1.4J	0.775J
55MW08	10-25	NI	3.1J	3.54	3.26J
55MW09	25-40	NI	384	590	509
55MW10	8-23	NI	184	261	312
55MW11	24.5-39.5	NI	NI	243	336
55MW12	15-30	NI	NI	13.1	4.15J
55MW13	15.5-25.5	NI	NI	NI	<5
55MW14	25.5-40.5	NI	NI	1,090	1,370
55MW15	40.5-55.5	NI	NI	46	37.3
55MW16	15-30	NI	NI	5.46	3.32J
55MW17	7.5-22.5	NI	NI	177	164
55MW18	49-59	NI	NI	2.0J	<5
55MW19	49.5-59.5	NI	NI	1.7J	<5
55MW20	14.5-29.5	NI	NI	0.7J	<5
55MW21	25.5-40.5	NI	NI	NI	<5
55MW22	52.5-67.5	NI	NI	NI	<5

TABLE 1
 Summary of TCE Analytical Results
Naval Activity Puerto Rico

Well ID	Screened Interval (feet bgs)	TCE Concentration (µg/L)			
		July 2009 (Baseline)	November 2009 (Baseline - Phase I)	January/ February 2010 (1st Quarter & Baseline Phase II)	April 2010 (2nd Quarter & Baseline Phase III)
55MW23	28.5-43.5	NI	NI	NI	1.24J

Notes:

Bold values indicate concentrations in excess of the CAO of 22 µg/L.

Shaded rows are wells outside the target treatment area

^a Samples contained permanganate and are assumed to have no TCE

J = value was measured below its practical quantitation limit.

-- = not sampled

NI = not yet installed

ISCO Injection Results Summary

The ISCO pilot study was conducted in December 2009 to evaluate the effectiveness of injecting an oxidizing agent (potassium permanganate) in the subsurface at SWMU 55 for treating TCE in groundwater. Generally, physical distribution of the injection solution was easily attained. However, injection of a low volume of solution (less than 1,000 gallons) resulted in the detection of sodium permanganate (NaMnO₄) 14 feet from the injection location, implying the existence of a thin (3 feet or less), but highly permeable zone intersected by the injection and monitoring wells in the pilot study area. During the pilot study, while injecting only between 25 and 40 feet bgs, NaMnO₄ was detected in monitoring wells screened in both the shallow (10 to 25 feet bgs) and deep (25 to 40 feet bgs) treatment zones. Introduction of NaMnO₄ to the more shallow wells likely occurred due to an increased elevation of the water table during the injection. However, detection of NaMnO₄ at these locations, after the introduction of a relatively small injection volume, may imply that the injection fluid was dispersed in a few primary flow paths instead of uniformly distributed throughout the aquifer. It appears that a thin vertical zone of the aquifer was impacted at least 25 feet from a single injection point, but that this is not indicative of the radius of influence through the entire saturated thickness of the target volume. During the actual injection, NaMnO₄ was detected only at adjacent injection wells and monitoring well 7MW07, in the middle of the injection area (Table 2).

NaMnO₄ was also detected at monitoring well 7MW24 at 920 milligrams per liter (mg/L) 2 weeks after the injection conclusion, indicating rapid migration of NaMnO₄ through preferential pathways. Monitoring well 7MW24 is approximately 40 feet downgradient of the injection area. As shown in Table 2, the NaMnO₄ concentration within the injection area declined quickly after the injection and was generally less than 1,000 mg/L at all wells sampled within 45 days of injection. No NaMnO₄ was detected at the site in April 2010.

TABLE 2
 Sodium Permanganate Concentrations
 SWMU 55
 Naval Activity Puerto Rico

Date	Event	Time	55IW01	55IW02	55IW03	55IW04	7MW07	7MW24	7MW10	7MW21	7MW22	7MW23
12/3/2009	ISCO Injection	11:35	43	--	1	1	4,330	1	--	--	--	--
12/3/2009	ISCO Injection	16:20	1,764	--	671	3,226	8,660	0	--	--	--	--
12/4/2009	ISCO Injection	13:00	--	--	727	--	11,171	0	--	--	0	0
12/8/2009	ISCO Injection	16:00	--	--	360	--	8,480	0	--	--	0	0
12/15/2009	ISCO Injection	11:20	--	--	142	--	3,880	0	--	--	0	0
12/15/2009	ISCO Injection	15:00	--	--	88	--	3,320	0	--	--	0	0
12/16/2009	ISCO Injection	11:30	--	--	--	--	5,600	0	--	--	0	0
12/16/2009	ISCO Injection	15:30	--	--	--	--	6,040	0	--	--	0	0
12/17/2009	ISCO Injection	11:20	--	--	--	--	4,640	0	--	--	0	0
12/17/2009	ISCO Injection	16:00	--	--	--	--	5,020	0	--	--	0	0
12/18/2009	1-Day Post Injection	10:00	2,280	520	2,360	11,040	--	--	--	--	--	--
12/22/2009	1-Week Post Injection	--	1,640	2,200	6,880	3	2,000	72	0	0	0	0
12/29/2009	2-Week Post Injection	--	618	172	2,600	11	816	920	--	--	0	0
1/5/2010	3-Week Post Injection	--	--	--	1,880	160	--	640	--	--	--	--
1/6/2010	3-Week Post Injection	--	360	492	--	--	720	--	--	--	--	--
1/28/2010	30-Day Post Injection	--	50	52	658	--	280	--	--	--	--	--
2/2/2010	30-Day Post Injection	--	--	--	--	10	--	328	--	--	--	--
4/10/2010	120-Day Post Injection	0	0	0	0	0	0	0	0	0	0	0

Notes:

All concentrations are in mg/L.

-- = not sampled

0 = Sample was collected but no visible NaMnO₄.

TCE only = Sent to lab for TCE only analysis. No visible NaMnO₄.

Bench testing was conducted prior to the field work to evaluate the permanganate demand exerted by naturally occurring organic material in the aquifer. Typically, this demand is much greater than that exerted by contaminants in the aquifer and represents the majority of the chemical demand required to treat a site. For SWMU 55, the permanganate demand was found to be low throughout the vertical zone impacted during the injection, and permanganate was expected to remain in the treatment zone for TCE oxidation for at least 6 months. However, the persistence was only 60 to 90 days. Because the permanganate demand measured during the bench testing was low, the rapid decline in the permanganate concentration detected in the injection area over time is likely due to flushing of the highly soluble permanganate from the treatment area in highly permeable groundwater flow zones.

Post-Injection Performance Monitoring Results

Post-injection performance monitoring results for groundwater samples collected in January 2010 (first quarterly monitoring event, conducted 30 days post-injection), and April 2010 (second quarterly monitoring event, conducted 120 days post-injection) are compared to the baseline sampling event data (from groundwater samples collected in July and November 2009 and January, February, and April 2010) in this section. The baseline sampling results are summarized in the CSM Summary section above.

As summarized on Figures 5 through 7, the post-injection first quarterly monitoring event conducted in January 2010 demonstrated significant reduction in the TCE concentrations throughout the vertical interval of the injection zone. Additionally, the treated area extended to monitoring well 7MW24, approximately 40 feet downgradient of the injection area.

As summarized on Figures 8 through 10, the post-injection second quarterly monitoring event conducted in April 2010 demonstrated an order of magnitude decrease (or more) in the TCE concentrations at 7MW07, 7MW24, 55IW01, and 55IW03 was maintained compared to baseline data, although TCE concentrations had increased from the January 2010 results. Less pronounced decreases were detected at 55IW02 and 55IW04. Of the injection wells sampled, there appeared to be more rebound in the shallow zone. Data collected at 7MW23 indicates a slight decrease compared to July 2009 and January 2010 data, and could be typical fluctuation due to sampling methods, or may indicate that treated groundwater from the injection zone has migrated into the area.

Although the TCE concentration at 55IW01 was an order of magnitude less than the baseline concentration, it is still representative of significant rebound in the dissolved TCE at this location in a relatively short timeframe. The rate of rebound in the TCE concentration is indicative of TCE migration back into the treatment area, rather than TCE desorption near 55IW01. Based on these results, there is likely an additional source of TCE immediately north of the injection area, closer to, or underneath, the building pad.

Considering the limited permanganate persistence, the apparent flushing of permanganate from the source area, and the extent of TCE rebound detected at 55IW01 during the second quarterly monitoring event, ISCO does not appear to be the optimal choice for full-scale application at SWMU 55.

Recommended Path Forward

AGVIQ-CH2M HILL recommends a shift from the current ISCO-only program to address groundwater contamination to a combined approach, including excavation to address a possible shallow source zone north of injection well 55IW01, one additional ISCO application to rapidly reduce TCE mass in the 55IW01 source area, and a longer-term in situ bioremediation (ISB) using enhanced reductive dechlorination (ERD) to attain treatment goals. The early success of ERD at the TCE area at SWMU 54 indicates that it may be more effective than ISCO in remediating the lower levels of TCE that remain after excavation of shallow source area soils and an additional ISCO application.

The rapid rebound detected at 55IW01 suggests a possible TCE source just to the north of this injection well. AGVIQ-CH2M HILL recommends the installation of two groundwater monitoring wells in this area (Figure 11) to determine the presence and extent of this possible source area. According to standard protocol, photoionization detector readings will be collected from soils throughout the soil column during well installation to provide qualitative determination of the presence of TCE in unsaturated materials, as well the saturated zone. The greatest TCE concentration of 33,000 µg/L was measured at 55IW01, screened from 10 to 25 feet bgs. Therefore, the proposed wells will also be screened from 10 to 25 feet bgs.

The third post-injection quarterly monitoring event will be conducted as planned in July 2010. In addition to analysis for permanganate (if visibly present) and TCE, groundwater samples from select wells will also be analyzed for ISB parameters, including cis-1,2-dichloroethene, vinyl chloride, dissolved iron, dissolved manganese, sulfate, sulfide, total organic carbon, methane, ethene, ethane, and alkalinity.

Based on ISCO pilot study results and site conditions, we recommend the following full-scale treatment approach for SWMU 55:

1. The source area will be more clearly defined by installing two new groundwater monitoring wells upgradient of existing well 55IW01.
2. If high levels of TCE are confirmed immediately north of existing well 55IW01, an approximately 20-foot by 20-foot excavation will be completed to a depth of 15 to 20 feet bgs. The portion of the excavation extending below the water table will be backfilled with gravel only. The portion of the excavation above the water table will be backfilled with a 70/30 mixture of organic mulch and gravel. This backfilled excavation will provide an infiltration gallery for a two-step treatment of soils and groundwater surrounding and downgradient of the hot spot. Figure 12 provides a cross section of the proposed source area excavation, which will be converted into an infiltration gallery/bioreactor.
3. The first step of treatment will involve the injection of permanganate into the clean gravel backfill at the bottom of the infiltration gallery. Approximately 1,275 pounds of NaMnO₄ will be introduced to the infiltration gallery as a 10-gram-per-liter solution. Downgradient groundwater monitoring wells will be used to evaluate permanganate distribution and persistence. Based on earlier injections, it is expected that this permanganate will move quickly down through the source area and destroy higher levels of TCE residuals immediately beneath and downgradient of the infiltration

gallery. The infiltration gallery will allow ISCO and ISB agents to passively migrate from the removed source area, following the likely migration paths of the TCE as it migrated from the source area.

4. The second step of treatment will involve a longer-term injection and recirculation of organic substrate in a larger volume of TCE-contaminated groundwater. This will not begin until the permanganate has been consumed or has flushed out of the TCE source area. Two options are available for implementation of the ISB treatment using the bioreactor: a simple injection of emulsified vegetable oil into the bottom of the infiltration gallery, or a longer-term recirculation of groundwater through the mulch/gravel layer to create an in situ bioreactor. AGVIQ-CH2M HILL has designed and installed several in situ bioreactors for TCE hot spots; based on this experience, it is believed that this is a viable option for SWMU 55.
5. Groundwater from select monitoring wells within the treatment zone will be recovered at low-flow rates and pumped into the top of the bioreactor, where TCE will be removed via ERD. Recovered groundwater may be amended with emulsified vegetable oil, fructose, or other soluble organics to promote the ERD. Groundwater passing through the bioreactor is treated and also promotes the recirculation of soluble organics in the surrounding aquifer to promote more widespread ERD. As a "green" alternative, the recirculation pumps can be powered using solar energy and will require little to no maintenance. It is estimated that the bioreactor would operate for 3 to 5 years and would require semiannual monitoring.

When permanganate is no longer detected at site monitoring wells (estimated to be approximately 4 months after distribution), the infiltration gallery will then be converted to a bioreactor.

References

AGVIQ-CH2M HILL. 2009. *Pilot Study Work Plan for SWMU 55*. Prepared for Naval Facilities Engineering Command Southeast. January.

Baker Environmental, Inc. (Baker). 2005. *Final Corrective Measures Study Final Report for SWMUs 54 and 55*. Prepared for Department of the Navy, Naval Facilities Engineering Command Atlantic Division. August.

Figures



- ⊕ Injection Well
- Monitoring and Observation Well
- ⊞ Recovery Well
- - - Fence

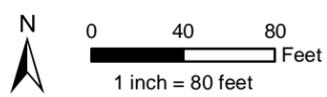
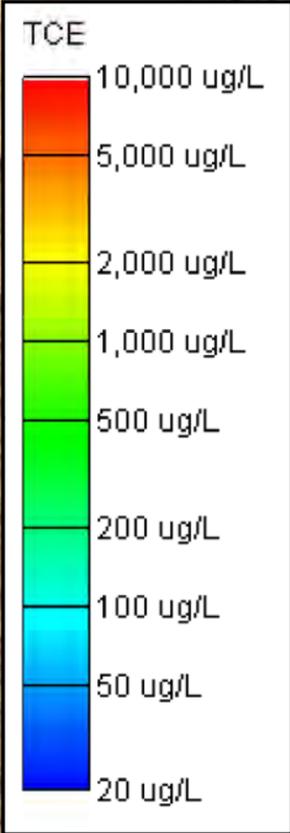
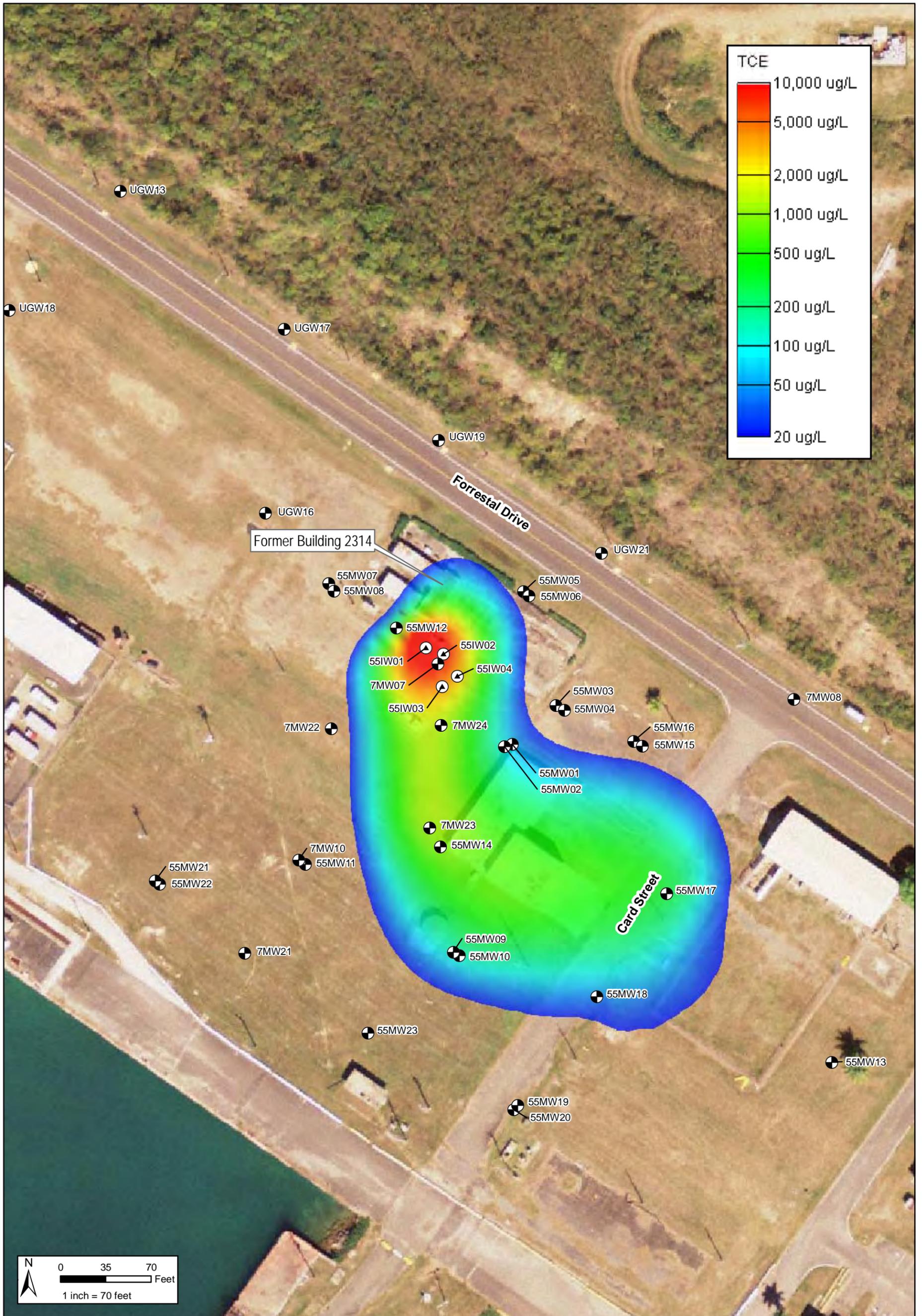
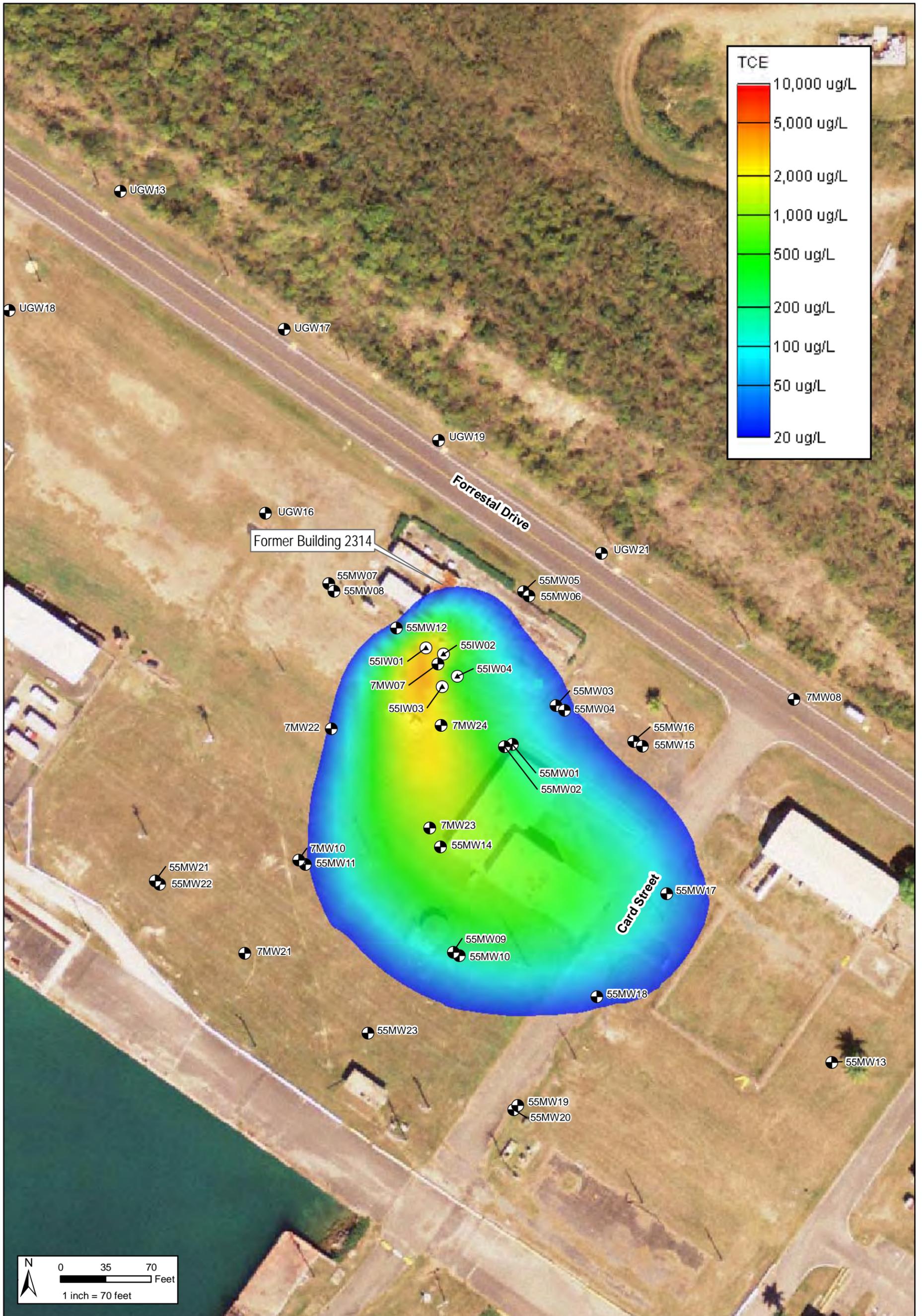


FIGURE 1
 SWMU 55 Monitoring and Recovery Well Site Map
 SWMU 55
 Naval Activity, Puerto Rico



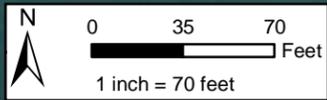
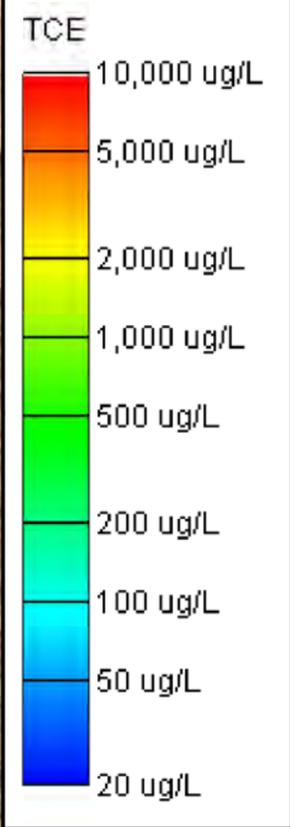
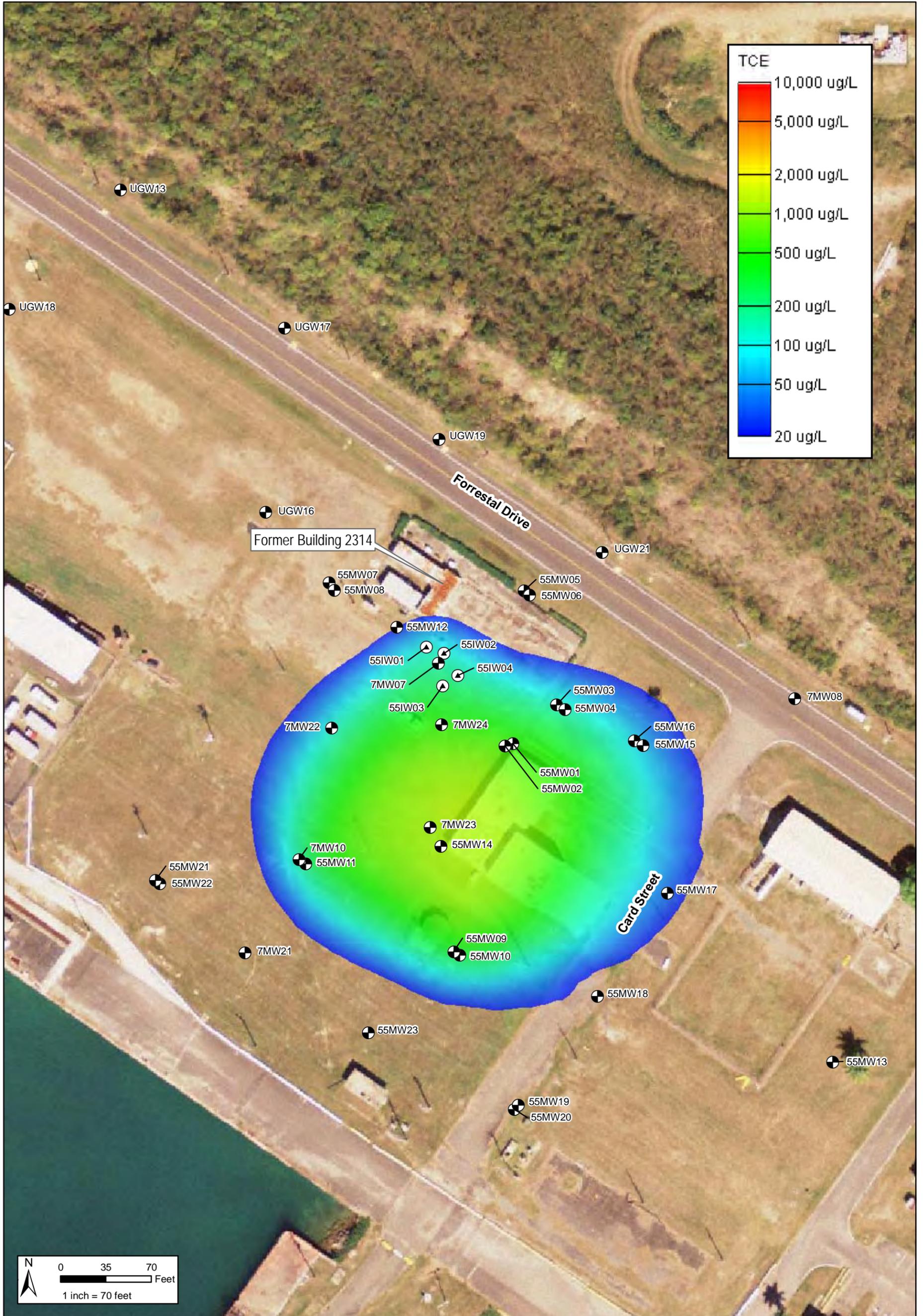
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 2
 TCE Concentration Approximately 14 ft bgs - Baseline
 SWMU 55
 Naval Activity, Puerto Rico



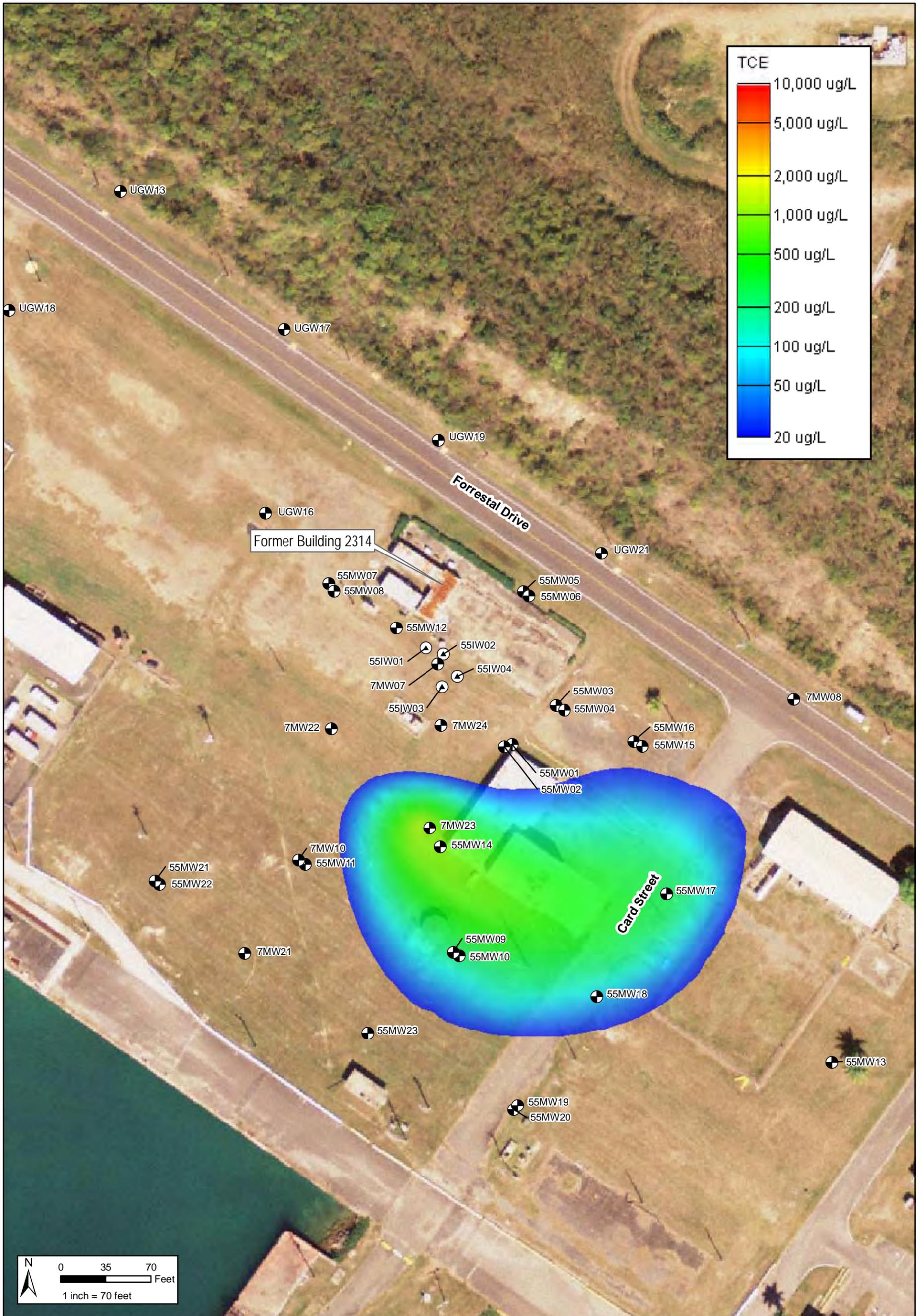
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 3
 TCE Concentration Approximately 25 ft bgs - Baseline
 SWMU 55
 Naval Activity, Puerto Rico



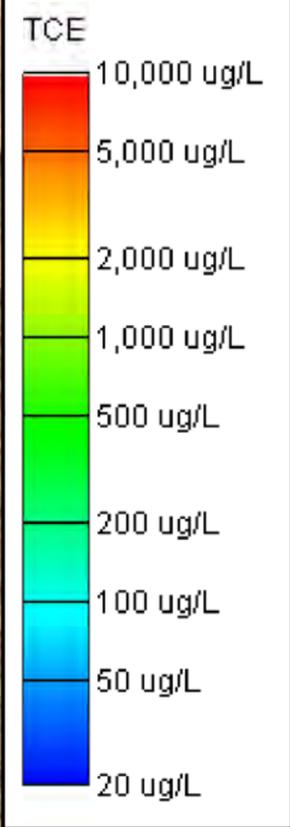
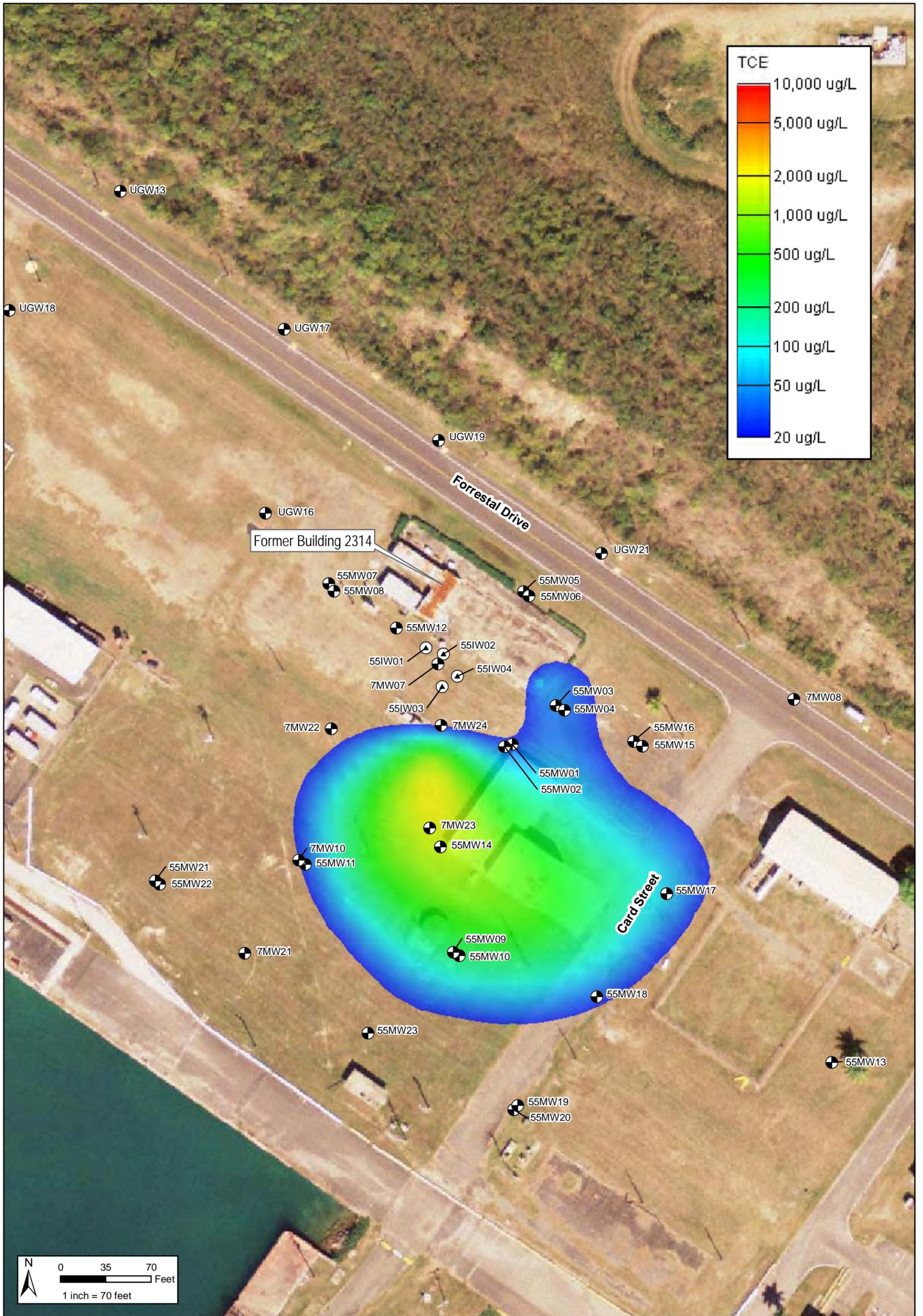
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 4
 TCE Concentration Approximately 41 ft bgs - Baseline
 SWMU 55
 Naval Activity, Puerto Rico



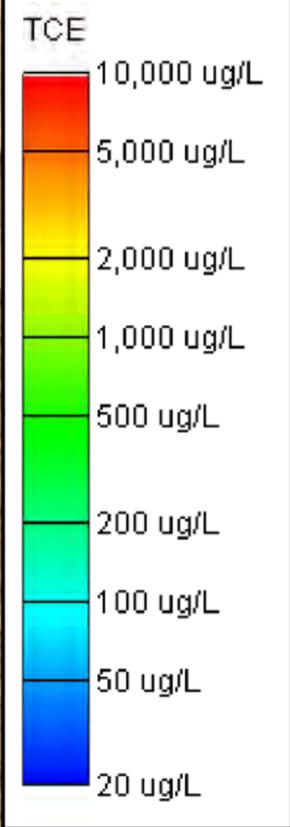
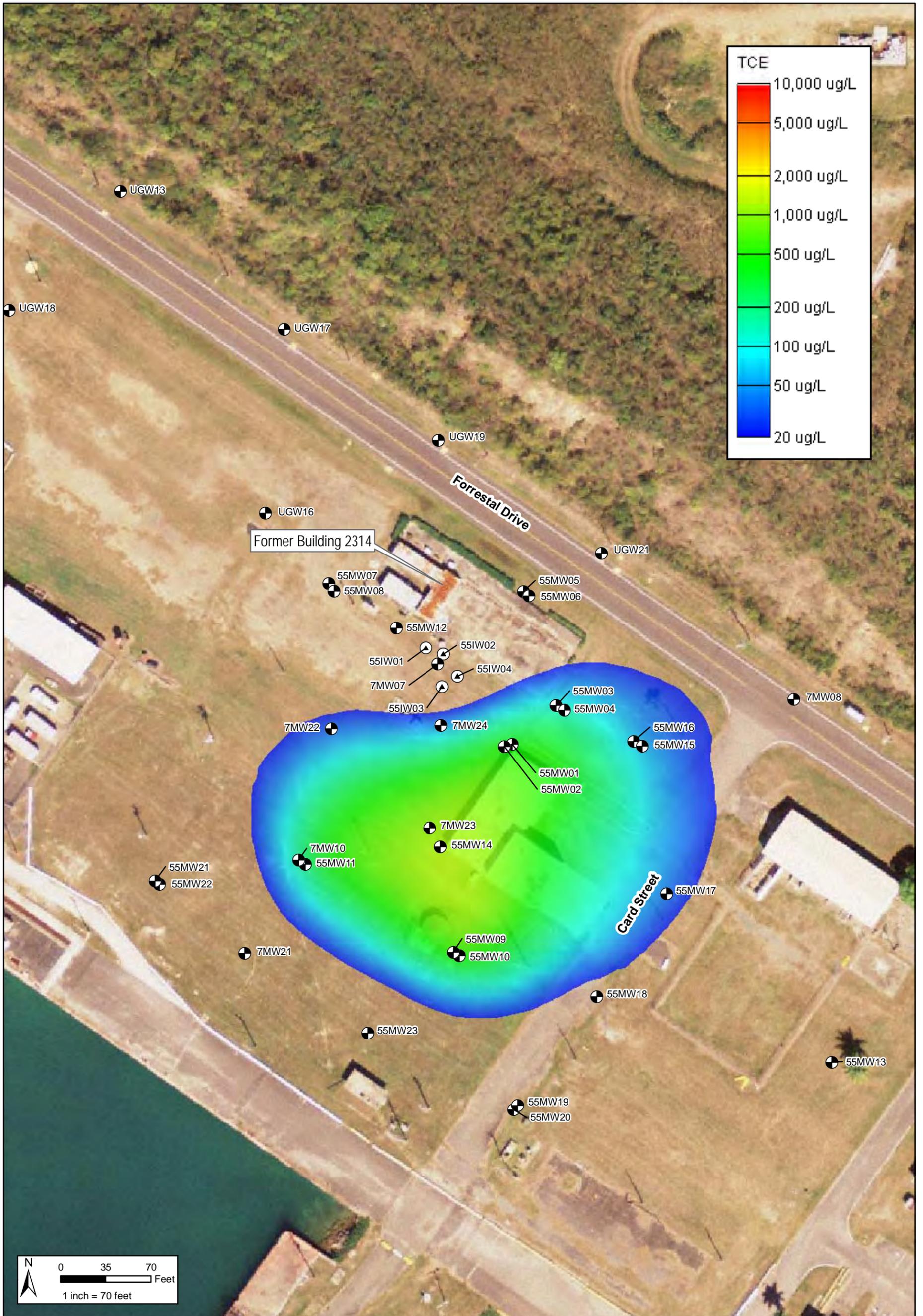
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 5
 TCE Concentration Approximately 14 ft bgs
 30 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico



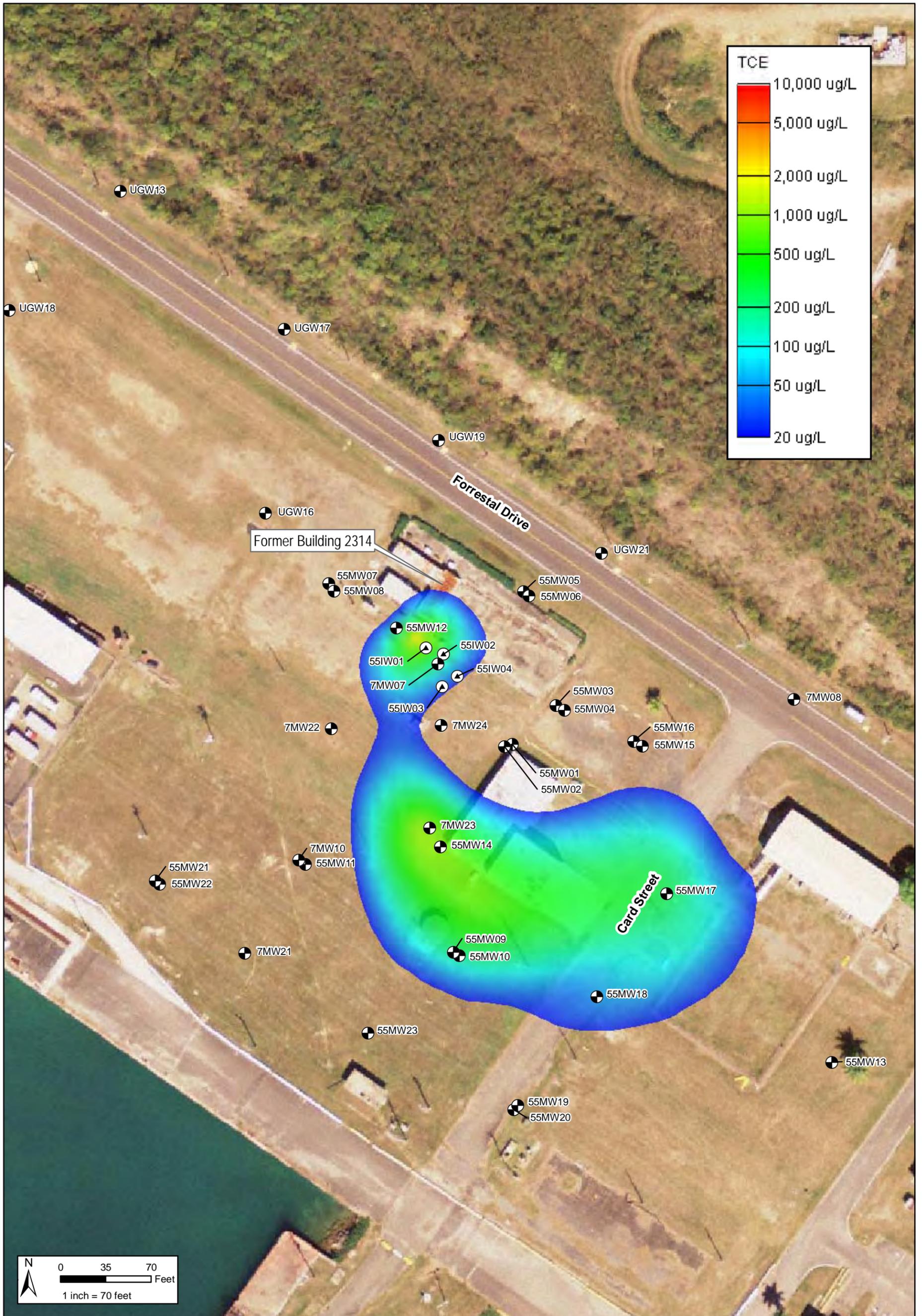
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 6
 TCE Concentration Approximately 25 ft bgs
 30 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico



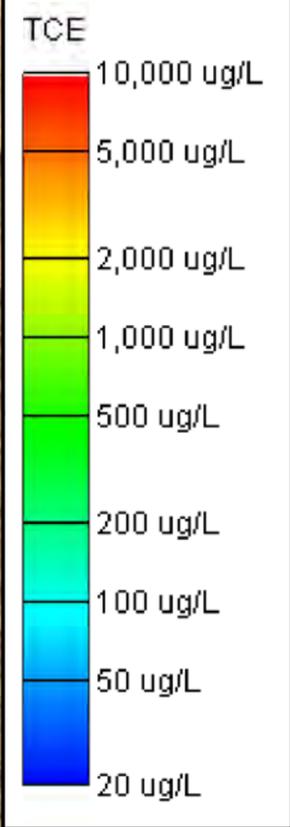
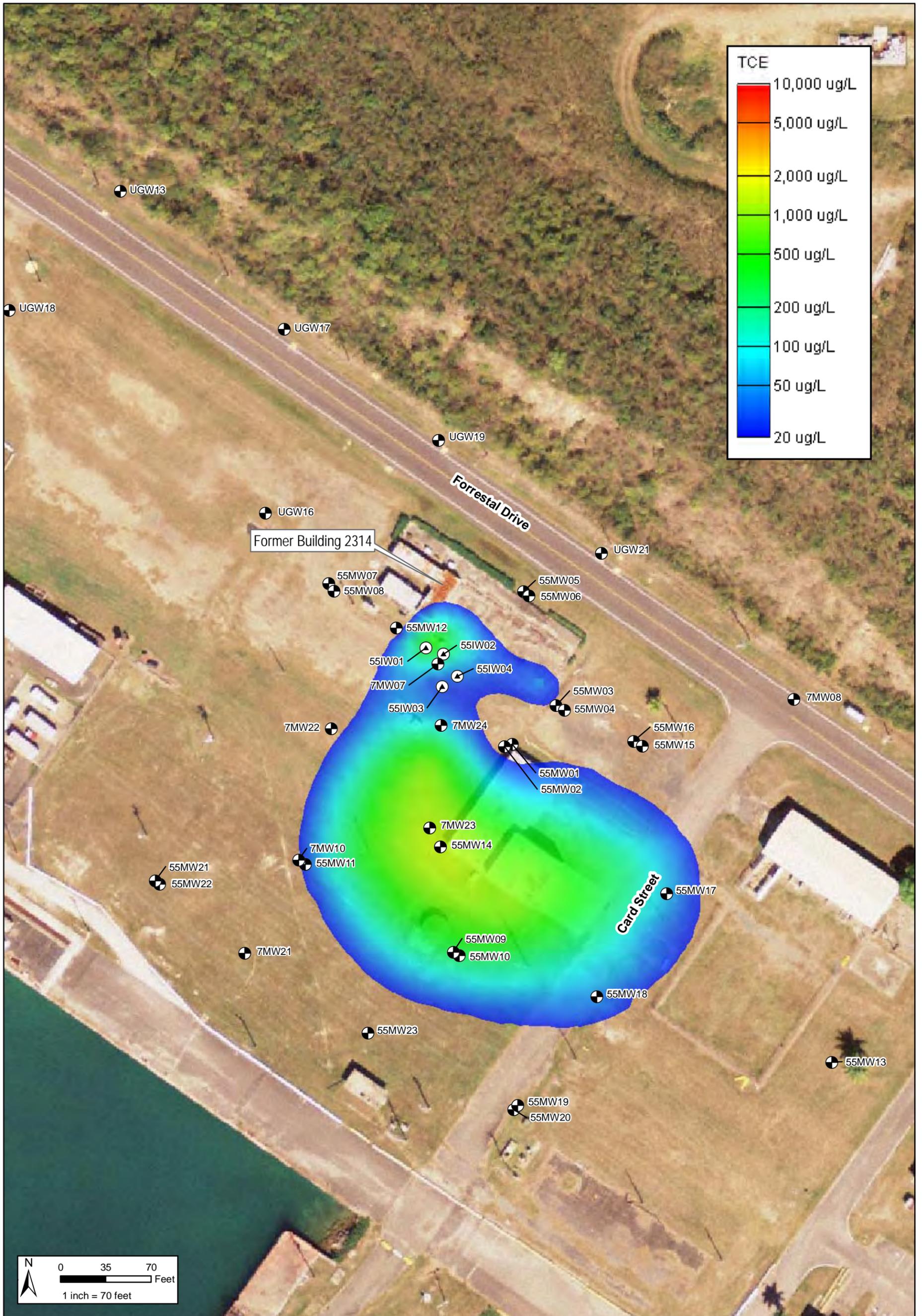
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 7
 TCE Concentration Approximately 41 ft bgs
 30 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico



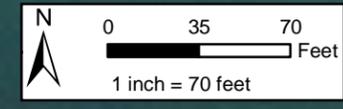
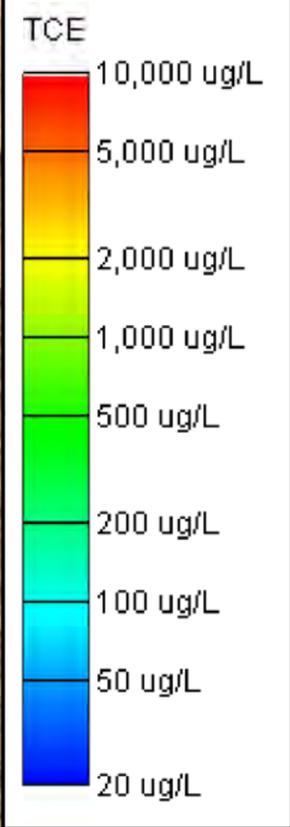
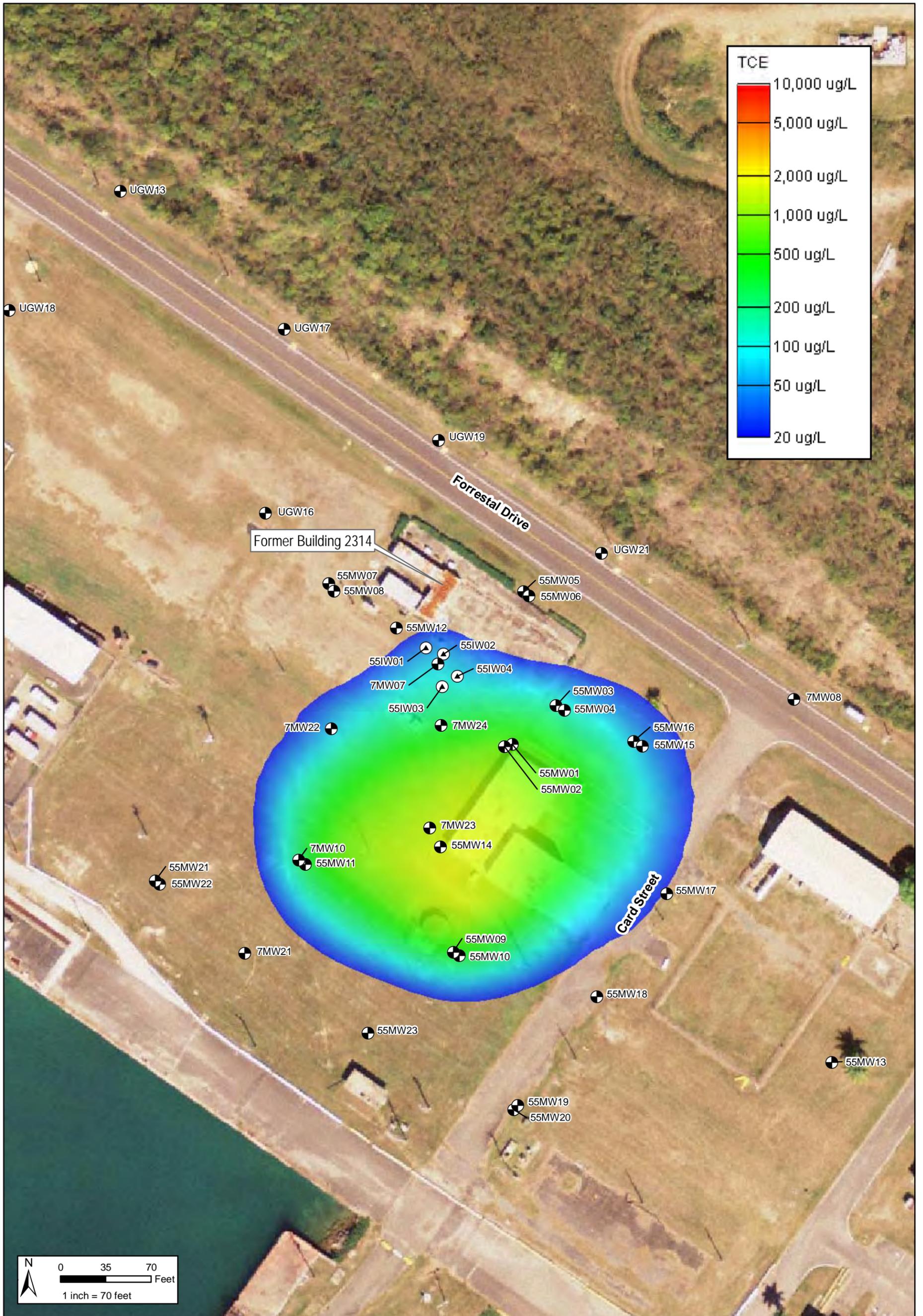
- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 8
 TCE Concentration Approximately 14 ft bgs
 120 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico



- Existing Monitoring Well Location
- ▲ Injection Well Location

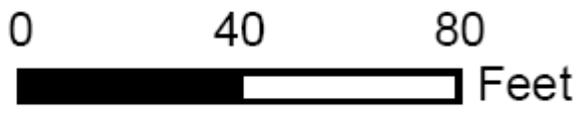
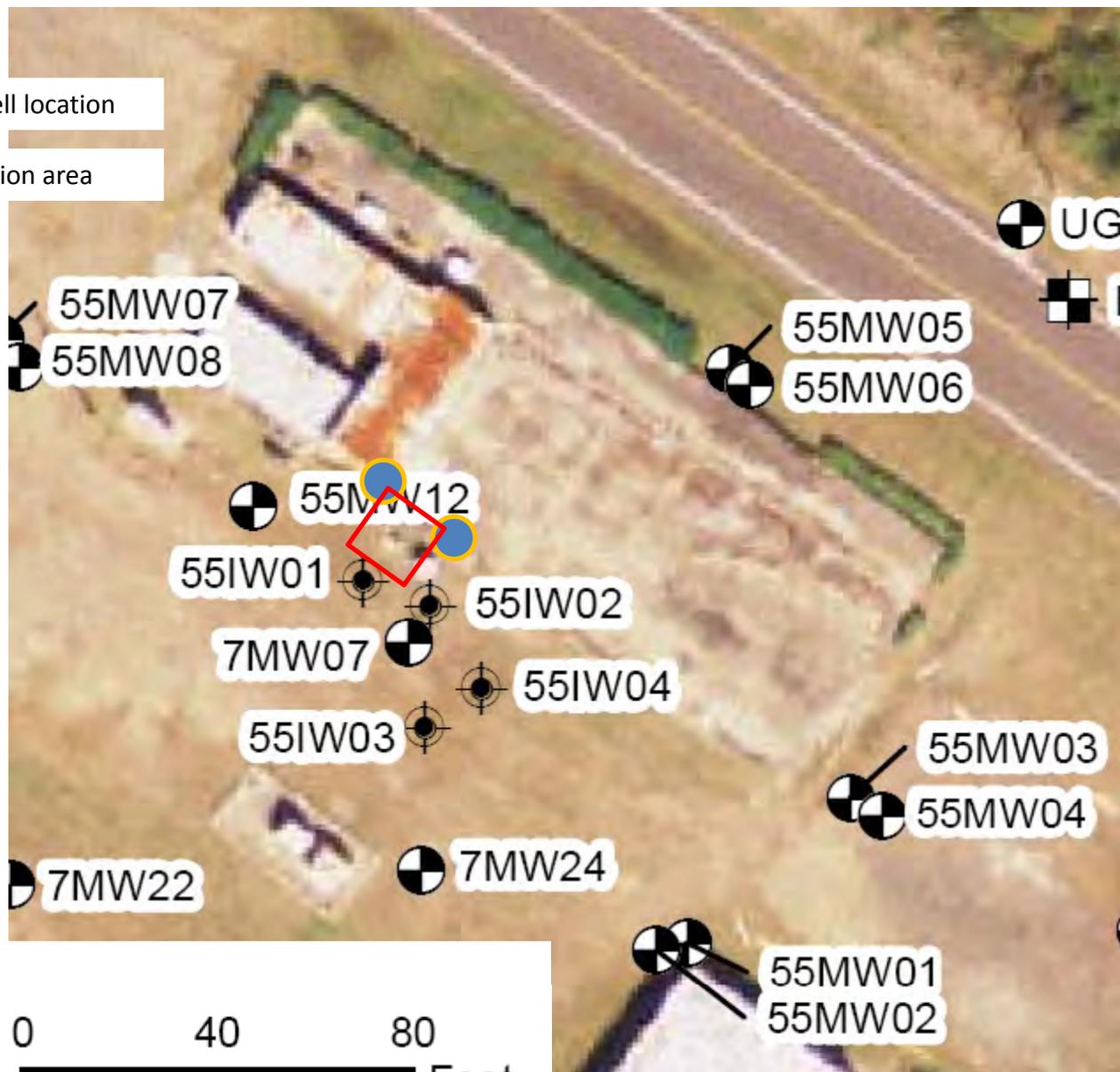
FIGURE 9
 TCE Concentration Approximately 25 ft bgs
 120 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico



- Existing Monitoring Well Location
- ▲ Injection Well Location

FIGURE 10
 TCE Concentration Approximately 41 ft bgs
 120 Days Post-ISCO Injection
 SWMU 55
 Naval Activity, Puerto Rico

- Proposed new well location
- Proposed excavation area



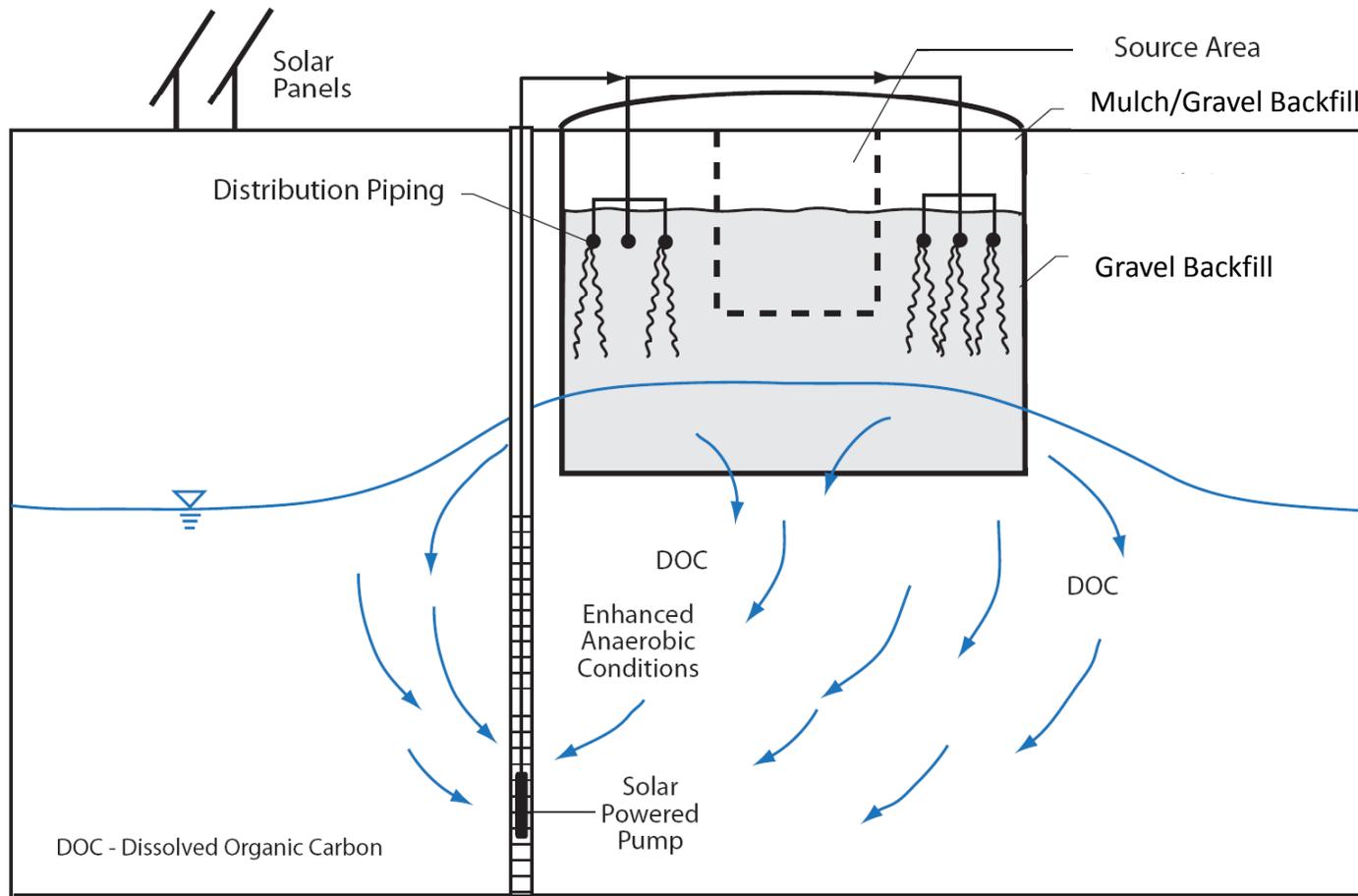


FIGURE 12
 Bioreactor Cross Section Schematic
 SWMU 55
 Naval Activity, Puerto Rico