Final

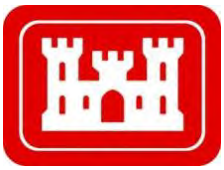
UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN (FIELD SAMPLING AND ANALYSIS PLAN)

PHASE I REMEDIAL INVESTIGATION AT CS-C503 PERFORMANCE-BASED RESTORATION JOINT BASE ANDREWS NAVAL AIR FACILITY WASHINGTON CAMP SPRINGS, MARYLAND

Contract W9128F-10-D-0025, DO #0002
DECEMBER 2012
VERSION: 00

Prepared for:

U.S. Air Force
11th CES/CEAN
3466 North Carolina Avenue
Joint Base Andrews, Maryland 20762-4803



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QAPP Worksheet #1 – Title and Approval Page

Uniform Federal Policy
Quality Assurance Project Plan
(Field Sampling and Analysis Plan)

Final
December 2012
Version: 00

Phase I Remedial Investigation at CS-C503
Performance-Based Restoration
Joint Base Andrews Naval Air Facility Washington
Camp Springs, Maryland

Prepared Under:
Contract Number: W9128F-10-D-0025
Delivery Order No. 0002

Prepared for:
U.S. Army Corps of Engineers – Omaha District

Prepared by:
Bay West, Inc.
5 Empire Drive
St Paul, Minnesota 55103
(651) 291-0456

Review Signature: _____

Rob Heimbach, PG
Site Lead, Bay West, Inc.

Review Signature: _____

John D. Verban
Quality Assurance Manager, Bay West, Inc.

Review/Approval Signature: _____

David Connolly
Restoration Chief, U.S. Air Force

Approval Signature: _____

Lucas Walsh
Project Manager, U.S. Army Corps of Engineers

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Acronyms and Abbreviations

%R .....Percent Recovery
µg/kg .....micrograms per kilogram
amsl .....above mean sea level
AFB .....Air Force Base
AFCEE .....Air Force Center for Engineering and the Environment
APP.....Accident Prevention Plan
Bay West.....Bay West, Inc.
bgs .....below ground surface
BTAG .....Biological Technical Assistance Group
BX .....Base Exchange
CA .....Corrective Action
CB .....Calibration Blank
CCV .....Continuing Calibration Verification
CERCLA .....Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR.....Code of Federal Regulations
CLP .....Contract Laboratory Program
CoC.....Chain of Custody
COPC.....Contaminant of Potential Concern
CPR .....Cardiopulmonary Resuscitation
CQC .....Contractor Quality Control
CQP .....Construction Quality Plan
CSM .....Conceptual Site Model
CS-C503 .....ERP designation for PCB-Contaminated Retention Pond
DERA .....Defense Environmental Restoration Account
DoD.....Department of Defense
DQCR .....Data Quality Control Report
DQI.....Data Quality Indicator
DQO.....Data Quality Objective
ECA.....External Certificate Authority
EICP.....Extracted Ion Current Profile
ERP.....Environmental Restoration Program
ERPIMS .....Environmental Resources Program Information Management System
FFA .....Federal Facility Agreement
FID.....Flame Ionization Detector
FOL.....Field Operations Lead
GC .....Gas Chromatograph
GPS .....Global Positioning System
H&S .....Health and Safety
JBA .....Joint Base Andrews Naval Air Facility Washington
IC .....Institutional Control
ICAL.....Initial Calibration
ICP.....Inductively Coupled Plasma
ICS.....Interference Check Solutions
ICV.....Second-source calibration verification
IDQTF .....Intergovernmental Data Quality Task Force
IDW.....Investigation-Derived Waste
ISTD .....Internal Standard
LCS Laboratory Control Sample
LCSD .....Laboratory Control Sample Duplicate
LOD .....Limit of Detection
LOQ .....Limit of Quantitation
MCLs .....Maximum Contaminant Levels
MD .....Maryland
MDE.....Maryland Department of the Environment
MDL .....Method Detection Limit
mg/Kg .....milligrams per kilogram
MS .....Mass Spectrometer
MS/MSD .....Matrix Spike/Matrix Spike Duplicate
NCP .....National Contingency Plan
NE.....None Established
NFA .....No Further Action
NFRAP .....No Further Remedial Action Planned
PAL.....Project Action Limit
PARCCS.....Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PCBs .....Polychlorinated Biphenyls
PDF .....Portable Document Format
PID.....Photoionization Detector

PGCHD ..... Prince George's County Health Department
pg/g ..... picograms/gram
PM ..... Project Manager
PPE ..... Personal Protective Equipment
PQL ..... Project Quantitation Limit
PQOs ..... Project Quality Objectives
QA ..... Quality Assurance
QAM ..... Quality Assurance Manager
QAPP ..... Quality Assurance Project Plan
QC ..... Quality Control
QL ..... Quantitation Limit
QSM ..... Quality Systems Manual
RI ..... Remedial Investigation
RPD ..... Relative Percent Difference
RPM ..... Remedial Project Manager
RSD ..... Relative Standard Deviation
RSLs ..... Regional Screening Levels
RTW ..... Retention Time Window
Site CS-C503
SOP ..... Standard Operating Procedure
SPCC ..... System Performance Check Compound
SSHO ..... Site Safety and Health Officer
SSHP ..... Site Safety and Health Plan
TAL ..... Target Analyte List
TBC ..... To Be Considered
TBD ..... To Be Determined
TCL ..... Target Compound List
TOC ..... Total Organic Carbon
TPH ..... Total Petroleum Hydrocarbons
UFP ..... Uniform Federal Policy
USACE ..... United States Army Corps of Engineers
USAF ..... United States Air Force
USEPA ..... United States Environmental Protection Agency
VI ..... Vapor Intrusion
VOA ..... Volatile Organic Analysis

Executive Summary

This site-specific Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) was developed by Bay West, Inc. (Bay West) with its teaming partners AMEC and Weston Solutions (from this point forward referred to as the Bay West Team) in response to a United States Environmental Protection Agency (USEPA) Region 3 request for federal facilities that have ongoing Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) sites to prepare UFP-QAPPs to cover any environmental data collection tasks that are being conducted for the United States Air Force (USAF) at Joint Base Andrews Naval Air Facility Washington (JBA) under contract number W9128F-10-D-0025 in accordance with the Federal Facility Agreement (FFA) (USEPA/USAF 2011). This UFP-QAPP is specific to the above-referenced contract number for the Multi-Site Remediation Performance-Based Restoration and is site-specific to CS-C503. CS-C503 has previously been referred to in other historical reports as the polychlorinated biphenyl (PCB)-Contaminated Sediment in the Retention Pond.

JBA is located in Prince George's County, Maryland (MD), near the community of Camp Springs, MD. Washington, D.C., is located approximately five miles northwest of the base. The principal features of the base occupy approximately 4,300 acres and consist of runways, airfield operations, an industrial area, housing, and recreational facilities.

JBA was originally established as the Camp Springs Army Air Field on August 25, 1942. The name was changed to Andrews Air Force Base (AFB) in 1947 when the USAF was established as a separate military service. The base has served as headquarters at various times for the Continental Air Command, the Strategic Air Command, the Military Air Transport Service, and the Air Force Systems Command. The current major tenant command is the Andrews Naval Air Facility. The missions of the Andrews Naval Air Facility are flight operations and photographic reconnaissance. In 1992, Andrews AFB became an Air Mobility Command Base. In May 1999, Andrews AFB was added to the CERCLA National Priorities List. The National Superfund electronic database identification number for the base is MD0570024000. In 2009, the name of the base was officially changed to JBA Naval Air Facility Washington to more accurately reflect the joint nature of the missions and operations at the base.

CS-C503, as designated in the Environmental Restoration Program (ERP) at JBA, is a 50-foot-by-500-foot retention pond located near the intersection of Arnold Avenue and North Perimeter Road and northeast of Building 1889 as shown on **Figure 1** and **Figure 2**. Construction as-builts for the storm sewer pipe network within the drainage area were not available during the development of this UFP-QAPP; and, therefore, the pipe network was verified during the development of this UFP-QAPP.

Maintenance activities at the retention pond were completed in 2007 and construction as-built drawings for the retention pond maintenance project were obtained from the JBA Stormwater Manager. The as-built drawings detail the excavation of 870 tons of sediment over a 220-foot-by-35-foot area. PCB-contaminated sediment was detected at a maximum concentration of 300 parts per billion (ppb) during the waste characterization sampling for disposal requirements. Currently, the source and extent of PCB contamination is unknown. In addition to the PCB contamination, total petroleum hydrocarbons (TPH) was also detected at a maximum concentration of 640 parts per million (ppm).

In 2009, URS completed a Final Evaluation Report, Air Force Compliance Clean-Up Sites, Identification and Evaluation of Defense Environmental Restoration Account (DERA) Eligibility for Multiple Locations at JBA. The report concluded that there has been a release of PCBs at the site and it was determined to be DERA eligible. The JBA ERP April 2011 Meeting reported

that CS-C503 was a Compliance Site and PCB Aroclor 1254 and TPH were detected within the pond soil/sediment.

The objective of the work covered under this UFP-QAPP will be to further assess the presence or absence of the contaminants of potential concern (COPCs) within the existing retention pond and to investigate the drainage area of the retention pond to determine possible sources of the PCB-contamination. To accomplish these objectives, the Bay West Team will complete a Phase I Remedial Investigation (RI) using: historical records, storm sewer piping network information, and laboratory analysis of soil, sediment, surface water, and stormwater samples collected from locations shown in **Figure 4**. The work to be completed under this UFP-QAPP is outlined on Worksheet #11 and in the Decision Logic on Worksheet #17.

Samples will be analyzed for the COPCs including Gasoline Range Organics/Diesel Range Organics (GRO/DRO) and PCBs using USEPA Methods 8015C and 8082A, respectively. GRO/DRO analytical results will be compared to the June 2008 Maryland Department of the Environment (MDE) Interim Final Cleanup Standards as no USEPA Regional Screening Levels (RSLs) are listed for comparison. PCB analytical results will be compared to the most current USEPA RSLs/Maximum Contaminant Levels (MCLs) at the time the Phase I RI Report is written. If PCBs are detected in the soil/sediment above their respective RSLs, additional analysis of Dioxins/Furans by USEPA Method 8290A will be conducted on the sample with the highest PCB concentration. If a detection of a dioxin or furan occurs, it will also be compared to the most current USEPA RSL. Total Organic Carbon (TOC) in soil/sediment will also be reported in the Phase I RI report. All analytical results will also be compared to the USEPA Ecological Soil Screening Levels (EcoSSLs) (USEPA 2008), the USEPA Region 3 Biological Technical Assistance Group (BTAG) Screening Benchmarks (USEPA 2006c) for freshwater and freshwater sediment, and the Basewide Background Study Report (CH2M Hill 2004), if applicable.

The investigative work at CS-C503 detailed above will be completed as necessary to verify/update the conceptual site model (CSM) and to support no further action (NFA)/no further remedial action planned (NFRAP) or to establish data quality objectives (DQOs) for additional RI work.

This site-specific UFP-QAPP is provided to describe the sampling, analysis, quality assurance (QA), and quality control (QC) requirements for CS-C503 to achieve the above mentioned objectives. This UFP-QAPP was prepared in accordance with:

- The guidance provided by the Intergovernmental Data Quality Task Force (IDQTF) for preparing Uniform Federal Policy Quality Assurance Project Plans, Part 1 Manual, Final Version 1, March 2005 (IDQTF 2005), and
- Chemical laboratory data requirements as described in Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.2 (v4.2), (DoD 2010).

This UFP-QAPP will be formally reviewed by JBA, the U.S. Army Corps of Engineers (USACE) – Omaha District, the regional USEPA Federal Facilities Remedial Project Managers (RPMs), MDE, and other stakeholder personnel authorized to review UFP-QAPPs.

QAPP Worksheet #2 – UFP-QAPP Identifying Information

Site Name/Project Name: JBA/Performance-Based Restoration
Operable Unit: CS-C503
Contractor Name: Bay West, Inc. (Bay West)
Contract Number: W9128F-10-D-0025
Contract Title: Performance-Based Restoration for JBA
Delivery Order Number (optional): 0002

1. This UFP-QAPP was prepared in accordance with the requirements of the *Federal Facility Agreement between the United States Environmental Protection Agency, Region 3 and the United States Department of the Air Force under CERCLA Section 120 (Sections IV and X) (USEPA/USAF 2011), Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) (IDQTF 2005), USEPA Guidance on Systematic Planning using the Data Quality Objectives Process, USEPA QA/G-4, (USEPA 2006a), USEPA Requirements for Quality Assurance Project Plans, Reissue, (QA/R-5), (USEPA 2006b),* and *Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2* (DoD, 2010).

2. Identify regulatory program: National Contingency Plan (NCP); Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)

3. Site Specific UFP-QAPP and appropriate addendums must be approved by United States Environmental Protection Agency (USEPA) Region 3 Federal Facility Remedial Project Manager (RPM) or other USEPA regional Quality Assurance (QA) authority.

4. This UFP-QAPP is a site-specific UFP-QAPP.

5. List dates of scoping sessions that were held:

Scoping Session	Date
Project Scope correspondence with JBA	December 2011 – January 2012
Tier 1 Meeting	December 7, 2011
Scoping Conference Call	June 27, 2012

6. List dates and titles of any UFP-QAPP documents written for previous site work that are relevant to the current investigation.

Title	Date
None	

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

   USACE – Omaha District (Partner)

   USEPA, Region 3 (Regulator)

   MDE (Regulator)

   Prince George's County Maryland (Regulator)

8. Lead organization

   USAF – JBA ERP

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

   Not Applicable

QAPP Worksheet #2 – UFP-QAPP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	
2	Table of Contents UFP-QAPP Identifying Information	
3	Distribution List	
4	Project Personnel Sign-Off Sheet	
Project Organization		
5	Project Organizational Chart	
6	Communication Pathways	
7	Personnel Responsibilities and Qualifications Table	
8	Special Personnel Training Requirements Table	
Project Planning/Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	See **Figure 1, Figure 2**, **Figures 3A through 3E**, and **Appendix A** for Additional Background Information
11	Site-Specific Project Quality Objectives	See **Figure 4** for Sampling Locations and **Attachment 2**
12	Measurement Performance Criteria Table	
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Various in Administrative Record
14	Summary of Project Tasks	See **Figure 4** for Sampling Locations
15	Reference Limits and Evaluation Table	
16	Project Schedule/Timeline Table	See **Appendix B** for the Site-Specific Schedule
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	See **Figure 1** and **Figure 4** for Site Location and Sampling Locations
18	Sampling Locations and Methods/Standard Operating Procedure (SOP) Requirements Table Sample Location Map(s)	See **Figure 4** for Sampling Locations
19	Analytical Methods/SOP Requirements Table	
20	Field Quality Control Sample Summary Table	
21	Project Sampling SOP References Table Sampling SOPs	See **Appendix C** for Sampling SOPs and **Appendix D** for Field Forms
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	See **Appendix E** for Laboratory SOPs and **Appendix F** for Laboratory Qualifications, Analytical Laboratory Methods, TestAmerica Laboratories, Inc.

QAPP Worksheet #2 – UFP-QAPP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
24	Analytical Instrument Calibration Table	Analytical Laboratory Methods, TestAmerica Laboratories, Inc.
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Analytical Laboratory Methods, TestAmerica Laboratories, Inc.
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal	Worksheet #21
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody (CoC) Form and Seal	See **Appendix C** for Sampling SOPs and **Appendix D** for Field Forms
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	
Data Management Tasks		
29	Project Documents and Records Table	
30	Analytical Services Table Analytical and Data Management SOPs	Worksheet #21
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	
32	Assessment Findings and Corrective Action Responses Table	
33	QA Management Reports Table	
D. Data Review		
34	Verification (Step I) Process Table	DoD QSM v 4.2, October 2010 (DoD, 2010), USEPA Analytical Services Branch (ASB) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review, (2005), and the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (USEPA, 2008b).
35	Validation (Steps IIa and IIb) Process Table	DoD QSM v 4.2, October 2010 (DoD, 2010), USEPA Analytical Services Branch (ASB) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review, (2005), and the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (USEPA, 2008b).
36	Validation (Steps IIa and IIb) Summary Table	DoD QSM v 4.2, October 2010 (DoD, 2010), USEPA Analytical Services Branch (ASB) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review,

QAPP Worksheet #2 – UFP-QAPP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
		(2005), and the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (USEPA, 2008b).
37	Usability Assessment	

QAPP Worksheet #3 – Distribution List

Name of UFP-QAPP Recipients	Title/Role	Organization	Telephone Number (Office)	Telephone Number (Cell Phone)	E-mail & Mailing Address	Document Control Number (Optional)
Andrew Sochanski	RPM	USEPA Region 3 Federal Facilities Group	(215) 814-3370		Sochanski.Andy@epamail.epa.gov U.S. Environmental Protection Agency 1650 Arch St. (3HS11) Philadelphia, PA 19103-2029	
Rick Grills	RPM	MDE	(410) 537-3398		rgrills@mde.state.md.us Maryland Department of the Environment Land Restoration Program 1800 Washington Blvd., Suite 625 Baltimore, MD 21230-1719	
Kenneth Clare	RPM	Prince George's County Health Department (PGCHD)	(301) 883-7689		KAClare@co.pg.md.us Prince George's County Health Department Largo Government Center 9201 Basil Court Largo, MD 20774	
Lucas Walsh	Project Manager (PM)	USACE	(402) 995-2750	(402) 350-3609	Lucas.V.Walsh@usace.army.mil CENWO-PM-HB Army Corps of Engineers, Omaha District 1616 Capitol Avenue Omaha, NE 68102-4901	
Molly Maxwell	Project Chemist	USACE	(402) 995-2288		Molly.C.Maxwell@usace.army.mil Army Corps of Engineers, Omaha District 1616 Capitol Avenue Omaha, NE 68102-4901	
Jennifer Grimm	Project Geologist	USACE	(402) 995-2267	(402) 619-6502	Jennifer.J.Grimm@usace.army.mil Army Corps of Engineers, Omaha District 1616 Capitol Avenue Omaha, NE 68102-4901	
Lynn Jenkins	Project Risk Assessor/ Industrial Hygienist	USACE	(402) 995-2219		Lynn.M.Jenkins@usace.army.mil Army Corps of Engineers, Omaha District 1616 Capitol Avenue Omaha, NE 68102-4901	

QAPP Worksheet #3 – Distribution List (Continued)

Name of UFP-QAPP Recipients	Title/Role	Organization	Telephone Number (Office)	Telephone Number (Cell Phone)	E-mail & Mailing Address	Document Control Number (Optional)
David Connolly	Restoration Chief	USAF	(301) 981-1653		david.connolly@afncr.af.mil 3466 North Carolina Avenue Andrews AFB, MD 20762	
Lydia Plotz	PM	USAF	(678) 478-4917		lplotz@portageinc.com 11th CES/CEAN 3466 North Carolina Avenue Andrews AFB, MD 20762	
Shirley McMaster	PM	Bay West		(651) 341-3263	shirleym@baywest.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	
Brad Kulberg	Project Quality Control (CQC) Systems Manager	Bay West	(651) 291-3444	(651) 755-7688	bradk@baywest.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	
Rob Heimbach	Site Lead	Bay West	(651) 291-3476	(651) 338-9821	robh@baywest.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	
Doug Hickey	Health and Safety (H&S) Manager	Bay West	(763) 479-3214		dhickeymn@yahoo.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	
John D. Verban	Quality Assurance Manager (QAM)	Bay West	(651) 291-3492	(651) 285-1691	johnv@baywest.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	
Jim Hubbell	Field Operation Lead (FOL) /Site Safety and Health Officer (SSHO)	Bay West		(651) 238-9482	jimh@baywest.com Bay West 5 Empire Drive St. Paul, MN 55103-2867	

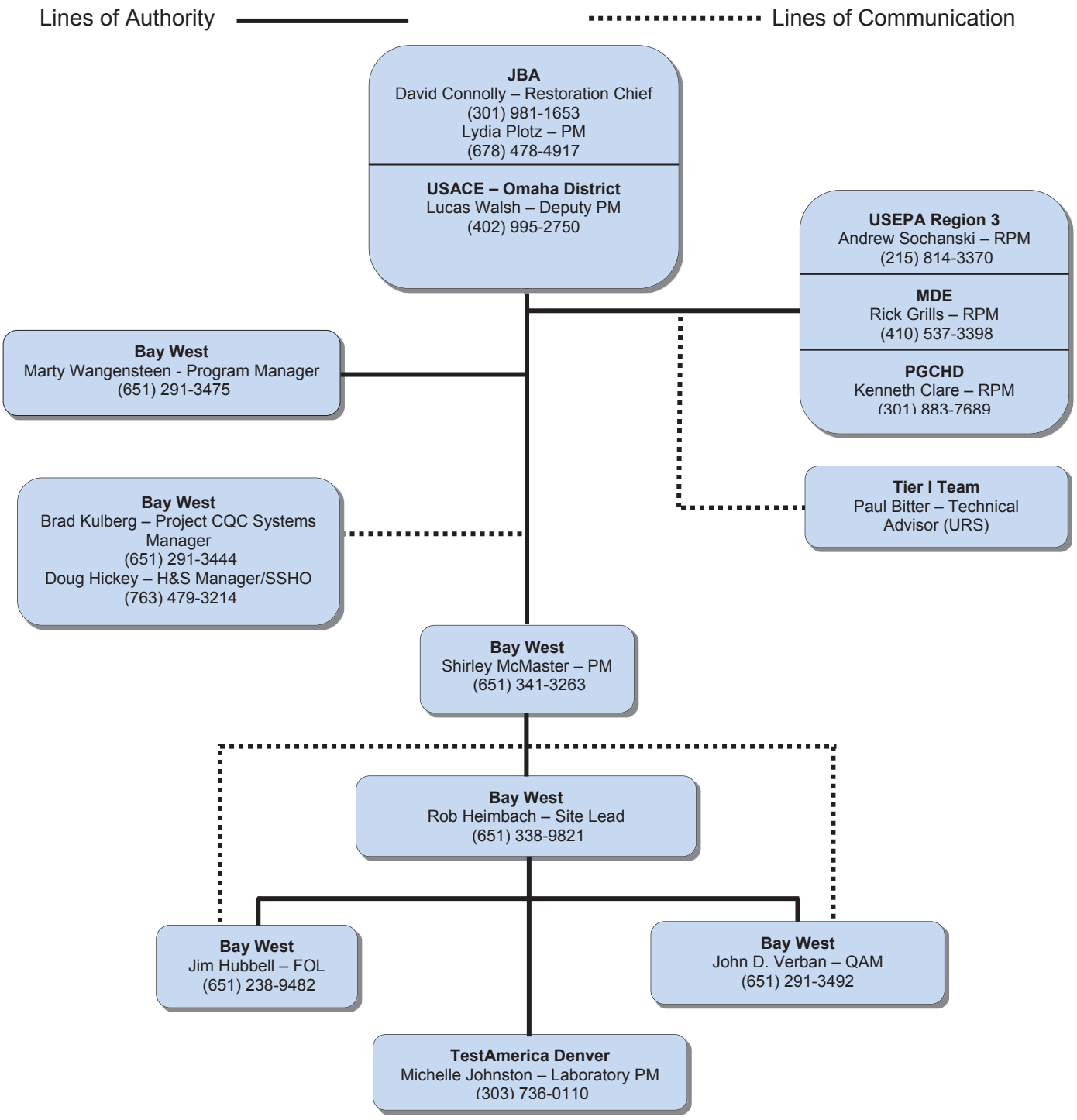
QAPP Worksheet #3 – Distribution List (Continued)

Name of UFP-QAPP Recipients	Title/Role	Organization	Telephone Number (Office)	Telephone Number (Cell Phone)	E-mail & Mailing Address	Document Control Number (Optional)
Michelle Johnston	Laboratory PM	TestAmerica Denver	(303) 736-0110		Michelle.Johnston@testamericainc.com TestAmerica Denver 4955 Yarrow Street Arvada, CO 80002	

QAPP Worksheet #4 – Project Personnel Sign-Off Sheet

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	UFP-QAPP Section Reviewed	Date UFP-QAPP Read
Andrew Sochanski	USEPA/RPM	(215) 814-3370		All	
Rick Grills	MDE/RPM	(410) 537-3398		All	
Kenneth Clare	PGCHD/RPM	(301) 883-7689		All	
Lucas Walsh	USACE/PM	(402) 995-2750		All	
Molly Maxwell	USACE/Project Chemist	(402) 995-2288		All	
Jennifer Grimm	USACE/Project Geologist	(402) 995-2267		All	
Lynn Jenkins	USACE/ Project Risk Assessor/ Industrial Hygienist	(402) 995-2219		All	
David Connolly	USAF/Restoration Chief	(301) 981-1653		All	
Lydia Plotz	USAF/PM	(678) 478-4917		All	
Shirley McMaster	Bay West/PM	(651) 341-3263		All	
Brad Kulberg	Bay West/Project CQC Systems Manager	(651) 291-3444		All	
Rob Heimbach	Bay West/Site Lead	(651) 338- 9821		All	
John D. Verban	Bay West/QAM	(651) 291-3492		All	
Jim Hubbell	Bay West/FOL/SSHO	(651) 238-9482		All	
Michelle Johnston	TestAmerica Denver/ Laboratory PM	(303) 736-0110		Worksheets 12, 15, 19, 23, 24, 25, 28, and 30	

QAPP Worksheet #5 – Project Organizational Chart



QAPP Worksheet #6 – Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Point of contact with the USACE, JBA, EPA, & MDE	Bay West PM Bay West Site Lead	Shirley McMaster Rob Heimbach	(651) 341-3263 (651) 338-9821	Overall management of the project. Maintains lines of communication between the USACE, USAF and MDE. Major field changes will be discussed and approved by the USACE PM prior to implementation.
Task Modification Request	Bay West FOL/SSHO	Jim Hubbell	(651) 238-9482	Immediately get approval from Bay West PM. Document via Task Modification Request form
UFP-QAPP Amendments	Bay West Site Lead	Rob Heimbach	(651) 338-9821	Send scope change to USACE PM within 30 days
Changes in Schedule	Bay West PM Bay West Site Lead	Shirley McMaster Rob Heimbach	(651) 341-3263 (651) 338-9821	Inform USACE and JBA PM via schedule impact letter as soon as impact is realized
Issues in the Field that Result in Changes in Scope of Field Work	Bay West FOL/SSHO Bay West PM Bay West Site Lead	Jim Hubbell Shirley McMaster Rob Heimbach	(651) 238-9482 (651) 341-3263 (651) 338-9821	Bay West FOL immediately informs Bay West PM; Bay West PM informs USACE and JBA PM within 24 hours; USACE PM issues scope change if warranted within 30 days; scope change to be implemented before work is executed
Recommendations to Stop Work and Initiate Work upon Corrective Action (CA)	Bay West FOL/SSHO Bay West PM Bay West Site Lead Bay West Project CQC Systems Manager Bay West H&S Manager JBA PM	Jim Hubbell Shirley McMaster Rob Heimbach Brad Kulberg Doug Hickey Lydia Plotz	(651) 238-9482 (651) 341-3263 (651) 338-9821 (651) 291-3444 (763) 479-3214 (678) 478-4717	Responsible Party immediately informs Bay West PM; Bay West PM will notify USACE and JBA PM
Analytical Data Quality Issues	Analytical Laboratory PM Bay West QAM Bay West PM	Michelle Johnston John D. Verban Shirley McMaster	(303) 736-0110 (651) 291-3492 (651) 341-3263	Immediately notify Bay West QAM, Project CQC Systems Manager, Bay West PM, and USACE Project Chemist if necessary

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)[1]
Andrew Sochanski	RPM	USEPA	EPA point of contact for the project, reviews all project related documents, and oversees regulatory compliance	Not Applicable
Rick Grills	RPM	MDE	MDE point of contact for the project, reviews all project related documents, and oversees regulatory compliance	Not Applicable
Kenneth Clare	RPM	PGCHD	PGCHD point of contact for the project, reviews all project related documents, and oversees regulatory compliance.	Not Applicable
Lucas Walsh	PM	USACE	USACE point of contact for project management tasks and reviews all project related documents.	Not Applicable
Molly Maxwell	Project Chemist	USACE	Project technical suggestions related to chemistry	Not Applicable
Jennifer Grimm	Project Geologist	USACE	Project technical suggestions related to geology	Not Applicable
Lynn Jenkins	Project Risk Assessor/Industrial Hygienist	USACE	Project suggestions related to risk assessment.	Not Applicable
David Connolly	Restoration Chief	USAF	USAF on base point of contact for the project, and reviews all project related documents.	Not Applicable
Lydia Plotz	PM	USAF	USAF site specific point of contact, and reviews all site specific documents.	Not Applicable
Marty Wangensteen	Program Manager	Bay West	Oversees project and responds to the U.S. Army Corps of Engineers, other stake holders	PE, PG, MS, Geology/ Civil Engineering, 24 years experience
Shirley McMaster	PM	Bay West	Oversee project, financial, schedule, and technical day-to-day management of the project.	PE, CQM, BS, Civil Engineering 38 years experience
Brad Kulberg	Project CQC Systems Manager	Bay West	Ensure quality aspects of the field sampling and analysis.	PMP, CQM, BS, Electrical Engineering 27 years experience
Rob Heimbach*	Site Lead	Bay West	Oversee technical tasks to ensure compliance with project objectives.	PG, BS, Geology 15 years experience

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table (continued)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)[1]
Doug Hickey	H&S Manager	Bay West	Overall responsibility for project H&S.	MS, Industrial Hygiene 29 years experience
Jim Hubbell*	FOL/SSHO	Bay West	Supervise, coordinate, and perform field sampling activities. Prepare geological interpretation and text.	BS Geology (in progress), CQM, UXO Tech I 23 years experience
John D. Verban*	QAM	Bay West	Participate in scoping, prepare lab scope, and coordinate with lab. Oversee data quality review and QA data validation deliverables.	BS, Chemistry, CQM 31 years experience
Michelle Johnston	Lab PM	TestAmerica Denver	Coordinate analyses with lab chemists, ensure the scope is followed, QA data packages, communicate with sampling contactor staff.	Resume located in laboratory
John Morris	Lab Quality Assurance Manager (QAM)	TestAmerica Denver	Oversee laboratory QA/QC program to insure laboratory and data reporting QA/QC requirements are achieved.	Resume located in laboratory

* Indicates Project Team Member
[1] Resumes are maintained at the Bay West corporate office and are available upon request.

QAPP Worksheet #8 – Special Personnel Training Requirements

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
Management and Technical Tasks	Construction Quality Management for Contractors	USACE	Renewed Every 5 Years	Management and Site Leads	Bay West	Bay West Project File
H&S/Chemical Sampling	40 hrs. /8-hr OSHA HAZWOPER [1] Refresher	Certified OSHA Trainer	40 hrs. - once 8-hr. - Annually	FOL/ Sampling Personnel	Bay West	Bay West Project File
H&S	8-hr OSHA Supervisory Training	Certified OSHA Trainer	Once	FOL	Bay West	Bay West Project File
Sampling, Sample Handling, documentation, and packing procedures	Technical employee training/on-site demonstration	Site Manager, lead sampler or designee	Prior to field work	FOL/ Sampling Personnel	Samplers, Bay West	Bay West Project File
First Aid	CPR[2] & First Aid	Certified CPR & First aid Trainer	Prior to field work	Site Manager/ Sampling Personnel	Samplers, Bay West	Bay West Project File

[1] Occupational Safety and Health Administration (OSHA) 29 *Code of Federal Regulations* (CFR) 1910.120.

[2] If there are two or more people on site, at least two on-site field technicians will maintain cardiopulmonary pulmonary resuscitation (CPR) and standard first aid training certificates. If only one worker is on site, that worker will maintain the above referenced certificates.

Safety requirements are addressed in greater detail in the Accident Prevention Plan, prepared under separate cover.

The selected analytical laboratory, TestAmerica, has successfully completed the laboratory evaluation process required as part of the Air Force Center for Engineering and the Environment (AFCEE) Quality Assurance Program and as described in DoD QSM v4.2 (DoD 2010). Copies of the current laboratory qualifications including current ELAP and MD certification in **Appendix F**.

QAPP Worksheet #9 – Project Scoping Session Participants Sheet

Project Name: Performance-Based Restoration **Projected Date(s) of Sampling:** October 2012 **Project Manager:** Shirley McMaster, Bay West	**Site Name:** JBA **Site Location:** Camp Springs, Maryland
Date of Session: December 2011 **Scoping Session Purpose: Site Coordination/Scope Approval**	

Name	Affiliation	Phone #	E-mail Address	Project Role
Andrew Sochanski	USEPA	(215) 814-3370	sochanski.andy@epamail.epa.gov	RPM
Rick Grills	MDE	(410) 537-3398	rgrills@mde.state.md.us	RPM
Kenneth Clare	PGCHD	(301) 883-7689	kaclare@co.pg.md.us	RPM
Luke Walsh	USACE	(402) 995-2750	Lucas.V.Walsh@usace.army.mil	PM
David Connolly	USAF	(301) 981-1653	david.connolly@afncr.af.mil	Restoration Chief
Roy Ellis	USAF	(301) 981-9623	roy.ellis@afncr.af.mil	PM
Shirley McMaster	Bay West	(651) 341-3263	shirleym@baywest.com	PM
Rob Heimbach	Bay West	(651) 291-3476	robh@baywest.com	Site Lead

Comments/Decisions:

Specific project scoping, proposed sampling approach and objectives were discussed with the JBA PM for CS-C503. A scoping summary was provided to the Tier 1 stakeholders during the December 2011 meeting. The investigative area and sampling scope has been discussed with JBA. It has also been decided that if COPCs are found in the initial round of sampling, revised sampling locations and matrix specific analytes will be provided to USACE and JBA in an updated figure.

Action Items:

Roy Ellis will review and provide any additional documents that might be applicable for this investigation.

Consensus Decisions:

Approach is consistent with other investigatory sites.

QAPP Worksheet #9 – Project Scoping Session Participants Sheet (Continued)

Project Name: Performance-Based Restoration **Projected Date(s) of Sampling:** October 2012 **Project Manager:** Shirley McMaster, Bay West	**Site Name:** JBA **Site Location:** Camp Springs, Maryland
Date of Session: June 27, 2012 **Scoping Session Purpose: Scoping Session (Conference Call)**	

Name	Affiliation	Phone #	E-mail Address	Project Role
Andrew Sochanski	USEPA	(215) 814-3370	sochanski.andy@epamail.epa.gov	RPM
Rick Grills	MDE	(410) 537-3398	rgrills@mde.state.md.us	RPM
Kenneth Clare	PGCHD	(301) 883-7689	KAClare@co.pg.md.us	RPM
Luke Walsh	USACE	(402) 995-2750	Lucas.V.Walsh@usace.army.mil	PM
Molly Maxwell	USACE	(402) 995-2288	Molly.C.Maxwell@usace.army.mil	Project Chemist
Jennifer Grimm	USACE	(402) 995-2267	Jennifer.J.Grimm@usace.army.mil	Project Geologist
Keith Freihofer	USAF	(301) 981-2337	keith.freihofer@afncr.af.mil	PM
Shirley McMaster	Bay West	(651) 341-3263	shirleym@baywest.com	PM
Rob Heimbach	Bay West	(651) 291-3476	robh@baywest.com	Site Lead

Comments/Decisions:

Specific project scoping, proposed area of concern, and proposed sampling approach were discussed with the JBA Tier I stakeholders to obtain a consensus on the sampling plan.

Action Items:

Bay West will revise the sampling locations per the scoping session discussions and submit the Draft UFP-QAPP to the regulators, JBA, and USACE.

Consensus Decisions:

The Tier I group reached a consensus on the CS-C503 sampling plan.

QAPP Worksheet #10 – Problem Definition

PCB-contaminated sediment (concentration of 300 ppb) was detected at site CS-C503 during maintenance activities associated with the retention pond in 2007 (TolTest 2008). To date, the source and extent of PCB contamination is unknown. In addition to the PCB contamination, TPH was also detected at a concentration of 640 ppm.

The background information in this worksheet summarizes information contained in the Administrative Record dated August 2011 for CS-C503, JBA, MD.

Site Location and Background

JBA is located in Prince George's County, MD, near the community of Camp Springs, MD. Washington, D.C. is located approximately five miles northwest of the base. The base occupies approximately 4,300 acres and consists of runways, airfield operations, an industrial area, and housing and recreational facilities.

JBA was originally established as the Camp Springs Army Air Field on August 25, 1942. The name was changed to Andrews AFB in 1947 when the USAF was established as a separate military service. The base has served as headquarters at various times for the Continental Air Command, the Strategic Air Command, the Military Air Transport Service, and the Air Force Systems Command. The current major tenant command is the Andrews Naval Air Facility. The missions of the Andrews Naval Air Facility are flight operations and photographic reconnaissance. In 1992, Andrews AFB became an Air Mobility Command Base. In May 1999, Andrews AFB was added to the National Priorities List. The National Superfund electronic database identification number for the base is MD0570024000. In 2009, the name of the base was officially changed to JBA Naval Air Facility Washington to more accurately reflect the joint nature of the missions and operations at the base.

CS-C503, as designated in the ERP at JBA, is a 50-foot by 500-foot retention pond located near the intersection of Arnold Avenue and North Perimeter Road and directly north of Building 1889. The elevations of the bottom and top of the retention pond are approximately 250 feet and 257 feet above mean sea level (amsl), respectively. In addition, the normal water level elevation is approximately 253 feet amsl resulting in a normal depth of water of 3 feet. **Figure 1** shows the location of CS-C503 within JBA. **Figure 2** presents the existing site features and recent aerial imagery.

As part of this Phase I RI, investigative work was conducted on June 6, 2012 with the goal of locating the storm sewer that outlets to the retention pond and identifying the storm sewer drainage area. This work was accomplished by contracting with Mid-Atlantic Utility Locating, who located and mapped the underground storm sewer network within the drainage area of the retention pond. The retention pond storm sewer originates in the vicinity of the Base Exchange (BX), Building 1811. Runoff from the building, asphalt loading area, and adjacent asphalt parking lot is conveyed via the storm sewer eastward from the BX building and then northward within the western boulevard of Arnold Avenue. The storm sewer eventually outlets at the south end of the retention pond.

History

A review of available historical aerial imagery identified modifications to landscape and structures at the CS-C503 site. The selected historical aerials are included on **Figure 3A** through **Figure 3E**. Available images predating 1964 were not included in this UFP-QAPP

QAPP Worksheet #10 – Problem Definition (Continued)

because they did not present any significant changes to the site during that timeframe. The following items were noted during the review:

- **1964** – Building 1889, The Club at Andrews, was constructed with adjacent parking lot to the South. North Perimeter Rd., Arnold Ave and Westover drive which surrounds Building 1889 were constructed.
- **1971** – Building 1889 was expanded to the north.
- **1974** – Building 1870, an electrical substation southwest of Building 1889, was constructed. There is a disturbed area south of Westover Drive and west of Arnold Avenue near the present day BX. Area is clear of trees and contains unidentified objects.
- **1982** – Previously mentioned disturbed area is clear of objects.
- **2000** – Building 1811, the BX, was constructed with an adjacent asphalt parking lot to the southwest. Retention pond northeast of Building 1889 was constructed.
- **2007** – Historical image for the site that pre-dates the 2007 reconstruction project which is described in the next section, Site Environmental History.

Based on the historical aerial image review, the retention pond was constructed between 1990 and 2000. Both the pipe inlet and overflow structure appear to have been constructed during this timeframe. The construction of Building 1870, the electrical substation, was noted because transformers have historically contained PCBs, and could be identified as a potential contaminant source; however, Building 1889, situated between the electrical substation and PCB-contaminated retention pond, would intercept potentially contaminated sediment/stormwater from the transformer site and the stormwater from the transformer site is directed to a separate storm sewer system. In addition, the aerial imagery review confirmed that Building 1889 pre-dates Building 1870; therefore, at no point was there a direct transport pathway between Building 1870 and the retention pond (CS-C503).

Site Environmental History

Environmental investigations have been conducted at the base since 1985 and are on-going under the USAF's ERP. The ERP was developed by the DoD in 1981 to identify, investigate, and clean up environmentally contaminated sites on military bases. CS-C503 was identified as a compliance restoration site through the ERP. To date, no removal actions or RIs have been completed at CS-C503.

The retention pond was re-constructed in 2007 with the objective of removing excess sediment, trees, and vegetation, and returning the site to a dry-bottom pond according to the Project Summary Report and associated construction as-builts (TolTest, Inc. 2008). The scope of work included excavating approximately 870 tons of sediment over a 220-foot-by-35-foot area. Prior to excavation, two composite samples were collected from the sediment in the retention pond for waste characterization and analyzed for VOCs, SVOCs, PCBs, pesticides, herbicides, metals, mercury, TPH, cyanide, sulfides, pH, and ignitability. The samples were collected from the influent and effluent areas of the pond. Results of the analysis indicated that PCB-contaminated sediment was present at concentrations of 190 ppb and 300 ppb. In addition, DRO was detected at concentrations of 120 ppm and 640 ppm. The waste profile was approved

QAPP Worksheet #10 – Problem Definition (Continued)

by MDE as non-hazardous. A total quantity of 870 tons of soil/sediment was excavated, temporarily staged along the east bank of the pond, and transported to the Brown Station Road Landfill in Prince George's County for use as daily cover material. Due to groundwater seeps encountered during excavation and following acceptance from the USAF and MDE, the site was restored to a wet-bottom pond (TolTest, Inc. 2008).

In 2009, URS completed a Final Evaluation Report, Air Force Compliance Clean-Up Sites, Identification and Evaluation of DERA Eligibility for Multiple Locations at JBA. The report concluded that there was a release of PCBs at the site based on the 2007 waste characterization sampling and it was determined to be DERA eligible. The JBA Environmental Restoration Program April 2011 Meeting reported that PCB Aroclor 1254 and TPH were detected within the pond soil/sediment at concentrations of 300 ppb and 640 ppb, respectively.

Problem Definition

To date, PCBs and TPH were detected in the soil/sediment at the CS-C503 retention pond and no additional analyses have been conducted with regards to the source or extent of contamination. The work covered under this UFP-QAPP includes the evaluation of the presence or absence of the COPCs within the existing retention pond and the evaluation of the associated drainage area for the presence or absence of COPCs.

The soil, sediment, surface water, and stormwater COPCs at CS-C503 are based on previous sampling results during the retention pond maintenance project and include the following:

- GRO/DRO
- Target Compound List (TCL) PCBs

In addition, if PCBs are detected above the screening criteria in the soil/sediment samples collected, one sample with the highest concentration of PCBs will be analyzed for dioxins/furans. These chemicals are associated with PCB manufacturing (as a chemical byproduct) in addition to the use of PCBs within transformers are sometimes oxidized to create dioxins and furans. TOC will also be reported on all soil and sediment samples to determine the organic carbon content of the samples.

To accomplish the above objectives, the Bay West Team will complete a Phase I RI. The objective of the Phase I RI will be to determine whether hazardous substances were released to the environment and/or whether hazardous substances have impacted the environment exceeding human health or environmental exposure criteria. The Phase I RI will include a Screening Level Human Health Risk Assessment as well as a Screening Level Ecological Risk Assessment to support potential recommendations of NFA/NFRAP, update the CSM, or to establish DQOs for additional RI work at CS-C503. The results will be presented to the Tier I partnering team for discussion.

Data Gaps

The above Phase I RI objectives were based on information available during the development of this UFP-QAPP. Previous sampling results detected both PCBs and TPH; however, the source and extent of contamination were not evaluated at that time.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

Project quality objectives (PQOs) define the type, quantity, and quality of data that are needed to answer specific environmental questions and support proper environmental decisions. To develop the PQOs, the DQO planning processes described in the USEPA "Guidance on Systematic Planning Using the Data Quality Objectives Process, USEPA QA/G-4" (USEPA, 2006a) are used. The USEPA QA/G-4 document suggests seven steps to be followed to develop project DQOs (performance and acceptance criteria) that clarify the study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support environmental decisions.

Step 1 of the DQO planning process, Problem Definition, is presented in Worksheet #10. Steps 2 through 7 are presented below.

Step 2 – Decision Statement

Field sampling data will be generated and analyzed from sampling events to be completed in 2012 in an effort to develop a Phase I RI at CS-C503. The following main decision points are anticipated during the RI:

- Are the COPCs present in the soil, sediment, surface water, and stormwater within the study area (See worksheet #10 for COPCs)?
- If COPCs are detected, do the specific compound concentrations exceed regulatory screening criteria, the USEPA Region 3 BTAG Screening Criteria, Basewide background levels, and EcoSSLs (See Step 5 – Analytical Approach)?
- If contamination exists, are there established pathways for potential exposure to humans or environmental receptors (See Step 5 – Analytical Approach)?
- What is the approximate extent of contamination, if any, both horizontally and vertically (See Step 3 – Decision Inputs)?
- Can the site be classified as NFA/NFRAP or do DQOs for additional RI work need to be established (See Step 5 – Analytical Approach)?

The field sampling data will be evaluated and used to summarize the existing conditions when developing the Phase I RI for the site. In addition, the information gathered from the sampling will be used by the USAF, in consultation with USACE, USEPA, and MDE, to evaluate the Phase I RI at CS-C503.

Step 3 – Decision Inputs

The objective of the following decision inputs is to answer the questions in Step 2. The Phase I RI will utilize a Triad-based decision framework with the following project tasks to achieve the above decision points:

- Summarize the available documentation surrounding the site (completed in May 2012);
- Map the existing stormwater piping network up-gradient of the existing retention pond to identify potential PCB-source areas (completed June 2012);
- Collect soil, sediment, and surface water samples within and adjacent to the retention pond to confirm the presence or absence of COPCs and assess potential contaminant

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

transport (sample locations and sample matrix at each location are provided on **Figure 4**); and

- Collect soil, sediment, and/or stormwater samples within the retention pond drainage area to evaluate the presence or absence of COPCs (sample locations and sample matrix at each location are provided on **Figure 4**). In addition, one additional soil sample will be collected in the woods southwest of the retention pond drainage area.

PCBs are relatively immobile in the environment due to their strong affinity to organic carbon and, therefore, PCB-contaminated soil is anticipated to be confined to the surface soils. In addition, since the 2007 retention pond maintenance work included the excavation of sediment within the ponding area, a 0- to 6-inch soil/sediment sampling depth is proposed during this investigation. The complete sampling procedures are detailed on Worksheet #14. The decisions made at this site will be in accordance with the Decision Logic included on Worksheet #17.

The soil, sediment, surface water, and stormwater samples collected at each location will be analyzed for the following COPCs by the USEPA Method identified:

- GRO/DRO by Method 8015C;
- TCL PCBs by Method 8082A; and,
- TOC by Method 9060A

If analytical results indicate the presence of PCBs above the RSLs in the soil/sediment, additional analysis for Dioxins/Furans by USEPA Method 8290A will be conducted on the sample with the highest PCB concentration. Samples will be sent to the TestAmerica Denver, Colorado, laboratory for GRO/DRO, PCBs, and TOC analysis. If Dioxin/Furan analysis is warranted, the sample of concern will be sent from TestAmerica Denver to TestAmerica West Sacramento for analysis. Real time decisions will be made in the field and communicated between the FOL and the Site Lead in conjunction with other JBA Phase I RI sites while laboratory analyses are being conducted. In addition, investigative efforts at other JBA sites included in the Performance-Based Restoration contract will be combined to minimize mobilizations and disruptions.

Copies of the analytical data will be stored at the laboratory for up to five years. Copies of analytical data will be copied to CD-ROM media. Bay West will upload the electronic copies of the data and reports to ERPIMS and will store them in its long term archive at the Bay West Headquarters office. Copies of the analytical data and the GIS source files will be included in the deliverables provided to USACE and JBA.

Project-specific measurement, sampling requirements, data management, validation criteria, USEPA RSLs, and MDE Cleanup Standards are presented on the following list of worksheets:

- Measurement Performance Criteria are described in Worksheet #12;
- Project documentation including the Phase I RI Report and recordkeeping is described in Worksheet #14;
- USEPA RSLs and MDE Cleanup Standards are presented in Worksheet #15;
- Sampling design and rationale are presented in Worksheet #17;

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

- Sample locations and sampling methods are listed in Worksheet #18, **Figure 4**, and Worksheet #14;
- Analytical group, methods, and requirements for sample bottleware, preservation, and holding times are listed in Worksheet #19;
- Sample custody and sample management is described in Worksheet #27;
- Data verification and validation are described in Worksheets #34 through #36; and,
- Data usability assessment is described in Worksheet #37.

Step 4 – Study Boundaries

The populations of primary interest are the areas directly adjacent to CS-C503 in which soil, sediment, surface water, and stormwater contain COPCs. Contaminant concentrations will be reported.

Step 5 – Analytical Approach

The analytical results will be compared to the following criteria as a screening tool during the Phase I RI to make recommendations for a NFA/NFRAP designation or for additional DQOs for future phases of work at this site:

- USEPA RSLs/MCLs for PCBs and Dioxins/Furans (the most current values at the time the Phase I RI Report is written) will be used as human health screening criteria;
- MDE Interim Final Cleanup Standards for GRO/DRO (MDE 2008) will be used as human health screening criteria;
- USEPA Region 3 BTAG Screening Benchmarks for Freshwater and Freshwater Sediment;
- USEPA Ecological Soil Screening Levels (EcoSSLs) (USEPA 2008) will be used to evaluate potential ecological receptors and pathways; and,
- Upper Tolerance Limits (UTLs) identified in the Basewide Background Study Report (CH2M Hill 2004) will be used to identify compounds that are naturally occurring within the installation boundaries.

Comparisons to the above criteria will made if found to be applicable based on the pathways and receptors identified in the screening level ecological risk assessment. For that reason, the USEPA RSLs and MCLs, as well as the MDE Cleanup Criteria are identified on Worksheet #15. If ecological pathways and receptors are identified, comparison to the most current screening criteria will be conducted. Concentrations exceeding the above screening criteria as well as their locations will be identified in the Phase I RI Report. No screening criteria are established for TOC. MDE cleanup standards for GRO/DRO are intended to be used in conjunction with a screening level risk assessment even though MDE considers these cleanup standards as “To Be Considered” (TBC) as no USEPA screening criteria exist.

Several compounds do not have RLs low enough to meet the USEPA EcoSSLs or USEPA Region 3 BTAG Screening Benchmarks. These compounds will be evaluated on a case-by-case basis and may not be compounds of concern at this particular site. If it is decided that the compounds are of concern at the site, the sampling and analytical methods will be evaluated. The laboratory will be contacted regarding options for preparation and analysis to lower the RL.

QAPP Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements (Continued)

A decision will then be made and documented as a path forward regarding the reporting of the compound(s) in question. In some cases, it may be impossible to meet the regulatory criteria. The lowest possible RL will be reported and noted in the data validation and the site report.

In order to be able to proceed to a NFA/NFRAP decision, the data must be of sufficient quality to justify the decision as determined by the project stakeholders. The analytical results from samples collected at CS-C503 will be provided at a USEPA Tier IV detail and will be validated to provide a Step IIb Validation Report, as detailed on Worksheet #36. The Tier I partnering team will be updated regarding the adequacy of the data prior to a NFA/NFRAP or additional DQO recommendation.

If COPCs are detected above the screening criteria and a NFA/NFRAP designation cannot be achieved with the data collected during the Phase I RI scope of work, the sampling information may be used to make decisions about the need for further remedial investigation and to evaluate the need for a long-term sampling program at CS-C503.

Step 6 – Performance and Acceptance Criteria

The UFP-QAPP consists of the collection of samples for laboratory analysis, recording of field measurements, and conduct data analysis. Sufficient numbers of samples, appropriate analytical and field methods, and appropriate QA/QC protocols will be applied to minimize errors that may affect future use of the data and subsequent decision making. Measurement and Performance criteria will be determined for each matrix, analytical group, concentration level, and analyte, as applicable. The criteria will relate to the parameters of: Precision, Accuracy/ Bias, Representativeness, Comparability, Completeness and Sensitivity (PARCCS). The parameters indicate the qualitative and quantitative degree of quality associated with the measurement data and are referred to as Data Quality Indicators (DQIs).

The Site Construction Quality Plan (CQP) has been included in **Attachment 2**. The objective of this CQP is to establish the project (QC systems that will ensure project activities are in conformance with project specifications. Bay West is responsible for the QC of work related to the performance of this contract. Bay West's QC system consists of the CQP, operations procedures, training, and a defined contractor quality control CQC organization.

Step 7 – Sample Design

The sample design for the planned sampling events in 2012 was developed in consultation with the federal, state, and local regulatory personnel that participate in the JBA Tier 1 partnering team. The sampling design and rationale are presented in Worksheet #17, and the sample locations and sampling methods are identified in Worksheet #18, on **Figure 4**, and in Worksheet # 14, respectively.

Data Management

The data will be reported on submittals from the lab and included in the Phase I RI Report. All data will be managed and archived by uploading analytical results into Environmental Resources Program Information Management System (ERPIMS).

QAPP Worksheet #12 – Measurement Performance Criteria Tables

Matrix Soil/Sediment/Water
Analytical Group GRO/DRO
Concentration Level Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance_Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SOP-1, SOP-9, SOP-11	SW 846 8015C, (3546), DRO: DV-OP-0006 DV-OP-0015, DV-GC-0027, GRO: DV-GC-0010	Precision – Overall	Relative Percent Difference (RPD) ≤ 30% when GRO/DRO detects for both field duplicate samples are ≥ Reporting Limit (RL).	Field Duplicates	S & A
		Precision - Lab	RPD ≤ 30% when GRO/DRO detects for both field duplicate samples are ≥ RL.	Laboratory Duplicates, Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD), Matrix Spike/Matrix Spike Duplicate (MS/MSD)	S & A
		Accuracy/Bias Contamination	No target compounds > ½ RL.	Equipment Blanks, Field Blanks, Method Blanks & Instrument Blanks	S & A
		Sensitivity	Within the laboratory's own in-house criteria as presented in the SOP or Quarterly LOQ Control Charts.	Low Level Check Standard at the RL.	A

[1]Reference number from QAPP_Worksheet_#21
[2]Reference number from QAPP_Worksheet_#23

QAPP Worksheet #12 – Measurement Performance Criteria Tables (Continued)

Matrix Soil/Sediment/Water
Analytical Group PCBs
Concentration Level Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance_Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SOP-1, SOP-9, SOP-11	SW 846 8082A, (3546), DV-OP-0006 DV-OP-0015, DV-GC-0021	Precision – Overall	RPD ≤ 30% when PCBs detects for both field duplicate samples are ≥ RL.	Field Duplicates	S & A
		Precision - Lab	QC acceptance criteria as specified by DoD QSM v4.2, Table G-17, p.G-17. If not specified in the tables, laboratory's own in-house criteria as presented in the SOP.	Laboratory Duplicates, LCS/LCSD, MS/MSD	S & A
		Accuracy/Bias	QC acceptance criteria as specified by DoD QSM v4.2, Table G-3 and G-17. If not specified in the tables, laboratory's own in-house criteria as presented in the SOP.	Surrogate Spikes, LCS, MS	A
		Accuracy/Bias Contamination	No target compounds > ½ RL.	Equipment Blanks, Field Blanks, Method Blanks & Instrument Blanks	S & A
		Sensitivity	Within the laboratory's own in-house criteria as presented in the SOP or Quarterly LOQ Control Charts.	Low Level Check Standard at the RL.	A

[1]Reference number from QAPP_Worksheet_#21
[2]Reference number from QAPP_Worksheet_#23

QAPP Worksheet #12 – Measurement Performance Criteria Tables (Continued)

Matrix Soil/Sediment
Analytical Group Dioxins/Furans
Concentration Level Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance_Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SOP-1, SOP-9	SW 846 8290A, WS-IDP-0005, WS-ID-0005	Precision – Overall	RPD ≤ 25% when Dioxins/Furans detects for both field duplicate samples are ≥ RL.	Field Duplicates	S & A
		Precision - Lab	QC acceptance criteria as specified by DoD QSM v4.2, Table F-6, p.F-33. If not specified in the tables, laboratory's own in-house criteria as presented in the SOP.	Laboratory Duplicates, LCS/LCSD, MS/MSD	S & A
		Accuracy/Bias	QC acceptance criteria as specified by DoD QSM v4.2, Table F-6, p.F-33. If not specified in the tables, laboratory's own in-house criteria as presented in the SOP.	LCS, MS	A
		Accuracy/Bias Contamination	No target compounds ≥ LOD for the analyte or ≥ 5% of the regulatory limit or ≥ 5% of the sample result, whichever is greater.	Equipment Blanks, Field Blanks, Method Blanks & Instrument Blanks	S & A
		Sensitivity	Within the laboratory's own in-house criteria as presented in the SOP or Quarterly LOQ Control Charts.	Low Level Check Standard at the RL.	A

[1]Reference number from QAPP_Worksheet_#21
[2]Reference number from QAPP_Worksheet_#23

QAPP Worksheet #12 – Measurement Performance Criteria Tables (Continued)

Matrix	Soil/Sediment
Analytical Group	TOC
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SOP-1 SOP 7	SW-846 9060A BR-WC-024	Precision – Overall	RPD ≤ 30% when SVOC detects for both field duplicate samples are ≥ RL.	Field Duplicates	S & A
		Precision – Lab	QC acceptance criteria as specified by DoD QSM. If not specified in the tables, laboratory's own in-house criteria.	Laboratory Duplicates LCS/LCSD MS/MSD	S & A
		Accuracy/Bias Contamination	No target compounds > 1/2 RL.	Equipment Blanks, Field Blanks, Method Blanks & Instrument Blanks	S & A
		Sensitivity	The laboratory's own in-house criteria as presented in the SOP.	Laboratory Fortified Blank at the RL.	A

[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data[1]	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Project Summary Report and Construction As-builts	TolTest, Inc. 2008. *Project Summary Report prepared for Implementation of Best Management Practices for Storm Water Point Source Control – Phase I. Andrews Air Force Base, Maryland.* July.	TolTest, Inc., Pond Maintenance Scope of Work, Construction As-builts, Waste Characterization Sampling Analytical Results.	Background information and previous sampling results.	Minimal limitations.

[1] Applicable historical reports are summarized on Worksheet #10.

QAPP Worksheet #14 – Summary of Project Tasks

The following presents a summary of the project tasks included in the Phase I RI:

- Mobilization/Demobilization
- Field Documentation
- Health and Safety
- Soil and Sediment Sampling
- Quality Control Samples
- Surveying
- Investigation-Derived Waste
- Data Review and Management
- Phase I RI Report Preparation

The subsequent sections detail the minimum requirements for the above project tasks. Additional Bay West SOPs are included in **Appendix C**.

Mobilization/Demobilization

Following the approval of the UFP-QAPP, Bay West will confirm the start date for the field work portion of the Phase I RI. Additionally prior to mobilization, Bay West will assemble the project team and complete the following tasks:

- Coordinate a utility location request with JBA 14 days prior to the commencement of the field activities. In addition, Bay West will submit a MD 811 Miss Utility locate request at least three days prior to the field work. If the utility locate personnel require someone from Bay West to be onsite during the locate, a project team member will be made available;
- Site access and site security procedures will be in accordance with the Security Plan under development for all Bay West Team project sites.
- Contact vendors and suppliers to coordinate goods and services required to support the field work (i.e. Investigation-Derived Waste [IDW] disposal facility, MD certified analytical laboratory, and a MD-licensed drilling subcontractor);
- Conduct a preparatory QC inspection. The purpose of this inspection is to ensure that all plans and procedures have been approved and that field personnel fully understand the procedures and methods used to safely perform operations at the site, individual duties and responsibilities, and all procedures associated with site operations; and,
- Conduct a field team orientation meeting to familiarize the team personnel with Site H&S requirements, the objectives and scope of field activities, and chain of command. This meeting will be attended by the field staff and the FOL/SSHO. The field team will be familiar with all sample locations and will identify related field support areas and requirements.

Following the initial project tasks described above, mobilization includes procurement of field equipment and supplies; mobilization of field staff, equipment, and supplies to the site; and site preparation. All equipment assemblies and calibrations, if necessary, will be performed prior to shipment to the site. Bay West mobilizations will be coordinated with the JBA PM a minimum of one week before the start of the field activities.

Once mobilized, site preparations will include:

- Survey the designated work area;

QAPP Worksheet #14 – Summary of Project Tasks (Continued)

- Identify resources/features that require protection during the field work;
- Locate material storage areas and temporary structures and sanitary facilities;
- Collect photographs of the area prior the field work;
- Stake the soil/sediment sample locations; and
- Setup the field sampling process and IDW storage areas.

Demobilization includes removing field equipment and supplies from the Site, returning rented equipment, managing IDW, performing general site cleanup, and organizing and finalizing field paperwork.

Field Documentation

All field records will be recorded using indelible ink in a permanently bound notebook with sequentially numbered pages. Sample IDs, locations, depth, descriptions, etc., will be described on field forms for each sample. Photo documentation of site activities will be completed in order to recreate field efforts and will be included in the final report. Additional information on field documentation and documentation sheets have been included on the Sampling SOPs included in **Appendix C.**

Additionally, the FOL/SSHO will complete a Daily Quality Control Report (DQCR) as referenced in the CQP in **Attachment 2**. A copy of the DQCR will be submitted to the Bay West PM and the USACE PM documenting the work performed each day field work is in progress. Copies of the DQCR will be available upon request.

Health and Safety

Prior to the initiation of any site work, all site workers will attend initial site-specific safety training, sign the Initial Site-Specific Safety Training Documentation form, complete a Medical Data Sheet, and attend all daily safety tailgate meetings. Records and logs of on-site safety meetings will be maintained. All site activities will comply with the Accident Prevention Plan (APP), which contains detailed information on site-specific health and safety requirements (**Attachment 1**).

Soil and Sediment Sampling

Soil and sediment samples will be collected and analyzed as outlined in the decision logic on Worksheet #17 to further evaluate the presence or absence of COPCs, if any, (outlined in Worksheet #11) at the CS-C503 site. Soil samples will be collected using a hand auger or a six-inch core sampler. Sediment samples will be collected with a sediment core sampler or an Ekman Bottom Sampler. Three sediment samples from the retention pond will be collected from the midline of the pond. An additional two sediment samples will be collected adjacent to the two southern midline pond samples along the west perimeter of the pond. Sampling personnel will wear waders while accessing submerged sediment sample locations. At each soil/sediment sample location, one sample will be collected to a depth of 6 inches. **Figure 4** shows the proposed soil and sediment sampling locations.

Additional information on soil and sediment sampling procedures is included in the Soil Sampling SOP (SOP-1) and Sediment Sampling SOP (SOP-9) included in **Appendix C**.

QAPP Worksheet #14 – Summary of Project Tasks (Continued)

Prior to completing sampling activities, the analytical parameters may be revised based on the decision logic flow chart provided on Worksheet #17. All stakeholders will be updated only if the proposed analytical parameters deviate from the list presented previously.

Soil Sampling Equipment Decontamination

A decontamination area will be set up adjacent to the work area for decontamination of site equipment. For personnel decontamination, the procedures will be conducted in accordance with the APP, "Personnel Decontamination." Personnel decontamination procedures to be used in the event of an emergency are outlined in the APP, "Decontamination During a Medical Emergency."

Surface Water and Stormwater Sampling

Surface water and stormwater samples will be collected from the retention ponds and the storm sewer system located at CS-C503. Surface water and stormwater samples will be collected from the most downstream location first, working in an upstream direction. Proposed sampling locations are provided on **Figure 4**.

The equipment required for surface water and/or stormwater sampling includes:

- Teflon dipper or disposable sampling scoop;
- Nylon rope;
- Dissolved oxygen, pH, temperature, conductance meters, and other instrumentation as required in the site-specific UFP-QAPP;
- Plastic sheeting;
- Appropriate sample bottles and temperature-controlled container;
- Five-gallon buckets with lids; and
- Shallow draft row boat, safety flotation vests (if necessary).

A representative sample for water quality testing will be collected at each sampling location that has sufficient water volume. The following sample collection procedures will be used to collect the surface water samples:

- When water and sediment samples are to be collected at the same location, the water sample will be collected first;
- All equipment will be protected from contact with foreign materials and contamination through use of clean plastic sheeting;
- Grab samples will be collected by directly submerging the sample container or by using a Teflon or Pyrex sampling device. Where the pond samples are being collected, the mouth of the sample collection device will be maintained completely under water and in an upstream direction;
- At the time of sampling, after collection of the sample for chemical analysis, a second sample will be collected for field measurement of temperature, pH, dissolved oxygen, conductivity, etc.;
- All samples will be preserved according to the requirements provided in this site-specific UFP-QAPP; and
- Samples containing anticipated high concentrations will be marked as such.

QAPP Worksheet #14 – Summary of Project Tasks (Continued)

Quality Control Samples

Field QC samples will be collected as part of the investigation, including field duplicates, equipment rinsate blanks, temperature blanks, and field blanks. Also, samples will be assigned and additional sample volume collected for the laboratory to perform matrix spike analysis. Additional quality control information is included in the Field Quality Control Samples SOP included in **Appendix C**.

Surveying

The location of utilities, structures, and sampling points will be noted in field books and/or on log sheets. Locations will be marked in the field with lath and documented with a combination of hand sketches and field-determined distances from surveyed benchmarks and reference points. Selected features, structures and all sample locations will be surveyed using a sub-meter handheld GPS unit and will be accurate to within 1 foot.

Investigation-Derived Waste

IDW is expected to be generated during the soil, sediment, surface water, and stormwater sampling and decontamination process

IDW will be managed so as to avoid additional degradation of the environment from which they are generated. IDW will be segregated into solids versus liquids, containerized, sampled for disposal categorization, and temporarily held at JBA while awaiting appropriate off-site disposal. Storage of IDW that has been determined to be hazardous by laboratory analysis will not exceed 90 days and will be stored at a location approved by the JBA PM. No on-site disposal of IDW will be performed nor will it be disposed of on the Base. Under no circumstances will water generated at one site be processed through a treatment system at another site.

Waste management requires categorizing the waste as hazardous or non-hazardous by USEPA Resource Conservation and Recovery Act definition. Categorization will include adequate sampling and analysis of the matrix to determine disposition of the material as hazardous or nonhazardous. The number of samples and the specific analyses required are based on the volume of the media, site contaminants, and the requirements of the disposal facility. Data validation will not be performed for IDW samples, as the analytical results will only be used for disposal purposes. Once the matrix has been properly categorized, it will be disposed of off-site at a permitted or authorized waste management facility. Waste disposal manifests and load tickets will be prepared and approved prior to transporting any contaminated soil off-base. The manifests will be provided to a JBA-authorized representative for review and signature. At this time, Keith Freihofer, Hazardous Materials Program Manager, JBA is designated to sign all shipping papers and/or waste manifests and Tim Hammond is an alternate signer.

Additional information with regards to IDW is provided in the Bay West SOP Investigation Derived Waste included in **Appendix C**.

Data Review and Management

Assessment/Audit Tasks

The assessment tasks reserved for this project include:

- Field Supervision;

QAPP Worksheet #14 – Summary of Project Tasks (Continued)

- Project Supervision;
- Field Sampling System Audit; and,
- Laboratory System Audit.

Worksheets #31 and #32 of this UFP-QAPP include details on the assessments and their respective CAs.

Data Management Tasks

Soil, sediment, surface water, and stormwater results will be compiled and presented in a comprehensive format to assist in the final evaluation and interpretation of results. All analytical data will be submitted to the ERPIMS ERPToolsX version 5.0 in the data format described in AFCEE Data Deliverable Requirements.

Data Review Tasks

Upon sample arrival, TestAmerica Denver will verify each sample's physical condition and ensure that all pertinent documentation associated with each sample is complete. Analytical data will be reviewed for completeness by the analytical laboratory. TestAmerica Denver staff will adhere to the verification process described in Worksheet #34 of this UFP-QAPP. Field data and pertinent documents (field logs, notes, and photographs) will be reviewed by Bay West personnel to establish the levels of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS), and usability of the final results with respect to the project DQOs.

Data Validation

Bay West will use Laboratory Data Consultants, Inc.'s Automated Data Review software to perform an automated data review equivalent to an USEPA Tier II evaluation and to provide preliminary discrete data qualification. During the full data validation, data qualifiers are appended to each result in the electronic data deliverables with validation criteria set at 100% of USEPA Tier III validation in accordance with the DoD QSM v4.2 for Environmental Laboratories (DoD 2010), the USEPA's Analytical Services Branch (ASB) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (USEPA 2011), and the USEPA's National Functional Guidelines for Superfund Organic Methods Data Review (USEPA 2008b). The USEPA Tier III validation is equivalent to the Step IIb validation detailed on Worksheet #35 of this UFP-QAPP.

Phase I Remedial Investigation Report Preparation

Bay West will complete a Phase I RI report to evaluate the presence or absence of COPCs at CS-C503. The Phase I RI report will include the following sections:

- *Introduction*. This section will describe the purpose and scope of the Phase I RI and summarize site background information, including site characteristics, description, and history.
- *Soil, Sediment, Surface Water, and Stormwater Sampling Results*. This section will include the COPCs detected, if any, as well as the location of any screening level exceedances.

QAPP Worksheet #14 – Summary of Project Tasks (Continued)

- *Screening Level Human Health Risk Assessment*. This section will include a comparison of the COPCs detected, if any, against the human health screening criteria identified in Worksheet #11.
- *Screening Level Ecological Risk Assessment*. This section will include a comparison of the COPCs detected, if any, against the ecological screening criteria identified in Worksheet #11.
- *Background Concentrations*. This section will include a comparison of the COPCs detected, if any, against the Basewide background concentrations identified in Worksheet #11.
- *Conclusions and Recommendations.* This section will include a discussion of the CSM, conclusions, and/or recommendations concerning site condition and further actions, including a NFA/NFRAP designation if applicable.

These reports will include all laboratory data, waste manifests, and daily field activity forms. Three versions (working copy, Draft, and Final) of each report will be submitted.

QAPP Worksheet #15 – Reference Limits and Evaluation

For CS-C503, the compounds will be compared to the USEPA RSLs/MCLs, except in the case of GRO/DRO which will be compared to the MDE Interim Final Cleanup Standards (MDE 2008). Worksheet #15 below contains reference tables of the complete GRO/DRO, PCBs, Dioxins/Furans compounds, and TOC as required for analysis by the DoD QSM v4.2. The full lists were provided as a resource for potential future use of these tables for subsequent expanded use of this UFP-QAPP for projects or sites requiring the analysis of other compounds or analytes. Where no regulatory criteria, i.e., cleanup or action level, is specified for the current ERP sites, the abbreviation NE was used to denote *none established*.

QAPP Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil/Sediment Samples, Gasoline Range Organic (GRO) and Diesel Range Organic (DRO) Analysis by Method 8015C

Analyte	CAS Number	Screening Criteria (mg/kg)	Screening Criteria Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific[2]	
					RLs (mg/kg)	MDLs (mg/kg)
Diesel Range Organics	68334-30-5	230	MDE Interim Final Guidance	4.0	4.0	0.678
Gasoline Range Organics	8006-61-9	230	MDE Interim Final Guidance	1.2	1.2	0.325

[1] The Screening Criteria Reference is taken from the MDE Interim Final Cleanup Standards (MDE 2008). MDE considers the use of these cleanup standards to be on a TBC basis, and not the primary-based cleanup criteria at CERCLA sites.

[2] Laboratory-specific MDLs and RLs are the limits that TestAmerica Denver can achieve when performing a specific analytical method. MDLs may be subject to update. These limits have been provided by TestAmerica Denver.

Matrix: Stormwater/Surface Water Samples, TCL GRO and DRO Analysis by Method 8015C

Analyte	CAS Number	Screening Criteria[2] (mg/L)	Screening Criteria Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific[3]	
					RLs (mg/L)	MDLs (mg/L)
Diesel Range Organics	68334-30-5	0.047	MDE	0.25	0.25	0.0326
Gasoline Range Organics	8006-61-9	0.047	MDE	0.025	0.025	0.01

[1]Reporting Limit for the highlighted analyte does not meet MDE Regulatory Criteria.

[2]The Screening Criteria Reference is taken from the MDE Interim Final Cleanup Standards (MDE 2008). MDE considers the use of these cleanup standards to be on a TBC basis, and not the primary risk-based cleanup criteria at CERCLA sites.

[3]Laboratory-specific MDLs and RLs are the limits that Test America Denver can achieve when performing a specific analytical method. MDLs may be subject to update and addenda will be issued in that case.

QAPP Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil/Sediment Samples, TCL PCBs Analysis by Method 8082A

Analyte	CAS Number	Screening Criteria (µg/Kg)	Screening Criteria Reference[1]	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific[2]	
					RLs (µg/kg)	MDLs (µg/kg)
PCB – 1016	12674-11-2	3,900	RSL - Residential	33	33	5.09
PCB – 1221	11104-28-2	140	RSL - Residential	33	33	15.6
PCB – 1232	11141-16-5	140	RSL - Residential	33	33	5.12
PCB – 1242	53469-21-9	220	RSL - Residential	33	33	9.12
PCB – 1248	12672-29-6	220	RSL - Residential	33	33	5.61
PCB – 1254	11097-69-1	220	RSL - Residential	33	33	5.52
PCB – 1260	11096-82-5	220	RSL - Residential	33	33	2.65

[1]The Screening Criteria Reference is taken from the USEPA RSL/MCL listing updated November 2011.

[2] Laboratory-specific MDLs and RLs are the limits that TestAmerica Denver can achieve when performing a specific analytical method. MDLs may be subject to update. These limits have been provided by TestAmerica Denver.

QAPP Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Stormwater/Surface Water Samples, TCL PCBs Analysis by Method 8082A

Analyte[1]	CAS Number	Screening Criteria (µg/L)	Screening Criteria Reference[1]	Project Quantitation Limit Goal (µg/L)	Laboratory-specific[2]	
					RLs (µg/L)	MDLs (µg/L)
PCB – 1016	12674-11-2	0.96	RSL – Tap Water	1	1	0.124
PCB – 1221	11104-28-2	0.0043	RSL – Tap Water	1	1	0.214
PCB – 1232	11141-16-5	0.0043	RSL – Tap Water	1	1	0.166
PCB – 1242	53469-21-9	0.034	RSL – Tap Water	1	1	0.104
PCB – 1248	12672-29-6	0.034	RSL – Tap Water	1	1	0.0915
PCB – 1254	11097-69-1	0.034	RSL – Tap Water	1	1	0.114
PCB – 1260	11096-82-5	0.034	RSL – Tap Water	1	1	0.16

[1]Reporting Limits for the highlighted analytes (7) do not meet Regulatory Criteria.

[1]The Screening Criteria Reference is taken from the USEPA RSL/MCL listing updated November 2011. The USEPA RSL/MCL used for the screening comparison during the Phase I RI report will be the most current version at that time.

[2] Laboratory-specific MDLs and RLs are the limits that Test America Denver can achieve when performing a specific analytical method. MDLs may be subject to update. Addenda will be issued in that case.

QAPP Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil/Sediment Samples, Dioxin/Furan Analysis by Method 8290A

Analyte	CAS Number	Screening Criteria (pg/g)	Screening Criteria Reference[1]	Project Quantitation Limit Goal (pg/g)	Laboratory-specific[2]		
					LOQs (pg/g)	LODs (pg/g)	MDLs[3]
2,3,7,8-TCDD	1746-01-6	4.5	RSL - Residential	1.0	1.0	0.15	EDL
1,2,3,7,8-PeCDD	40321-76-4	NE	NE	5.0	5.0	0.75	EDL
1,2,3,4,7,8-HxCDD	39227-28-6	NE	NE	5.0	5.0	0.75	EDL
1,2,3,6,7,8-HxCDD	57653-85-7	NE	NE	5.0	5.0	0.75	EDL
1,2,3,7,8,9-HxCDD	19408-74-3	NE	NE	5.0	5.0	0.75	EDL
1,2,3,4,6,7,8-HpCDD	35822-46-9	NE	NE	5.0	5.0	0.75	EDL
OCDD	3268-87-9	NE	NE	10	10	1.5	EDL
2,3,7,8-TCDF	51207-31-9	NE	NE	1.0	1.0	0.15	EDL
1,2,3,7,8-PeCDF	57117-41-6	NE	NE	5.0	5.0	0.75	EDL
2,3,4,7,8-PeCDF	57117-31-4	NE	NE	5.0	5.0	0.75	EDL
1,2,3,4,7,8-HxCDF	70648-26-9	NE	NE	5.0	5.0	0.75	EDL
1,2,3,6,7,8-HxCDF	57117-44-9	NE	NE	5.0	5.0	0.75	EDL
2,3,4,6,7,8-HxCDF	60851-34-5	NE	NE	5.0	5.0	0.75	EDL
1,2,3,7,8,9-HxCDF	72918-21-9	NE	NE	5.0	5.0	0.75	EDL
1,2,3,4,6,7,8-HpCDF	67562-39-4	NE	NE	5.0	5.0	0.75	EDL
1,2,3,4,7,8,9-HpCDF	55673-89-7	NE	NE	5.0	5.0	0.75	EDL
OCDF	39001-02-0	NE	NE	10	10	1.5	EDL

[1] The Screening Criteria Reference is taken from the USEPA RSL/MCL listing updated November 2011.

[2] Laboratory-specific LOQs and LODs are the limits that TestAmerica West Sacramento can achieve when performing a specific analytical method. MDLs may be subject to update. These limits have been provided by TestAmerica West Sacramento.

[3] Estimated Detection Limit (EDL) - For each chemical not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for nondetected chemicals rather than reporting the LOQ.

LOQ = Limit of Quantitation
LOD = Limit of Detection
pg/g = Picogram per gram

QAPP Worksheet #15 – Reference Limits and Evaluation Table (Continued)

Matrix: Soil/Sediment, Total Organic Carbon Analysis by Lloyd Kahn Method

Analyte	CAS Number	PAL (mg/kg)	PAL Reference	PQL Goal (mg/kg)	Laboratory-Specific (mg/kg)	
					RL[1]	MDL[2]
Total Organic Carbon	7440-44-0	NE	NE	1,000	1,000	110

[1] Quantitation limits are laboratory specific measurements but should approach the PQL goal and the lowest initial calibration standard will be run at the QL.
[2] MDL = method detection limit
mg/Kg = milligrams per kilogram

QAPP Worksheet #16 – Project Schedule / Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Working Draft UFP-QAPP	Bay West	October 2011	March 2012	UFP-QAPP to JBA and USACE	March 1, 2012
Draft UFP-QAPP	Bay West	April 2012	June 2012	UFP-QAPP to JBA, USACE, USEPA, and MDE	October 15, 2012
Draft Final UFP-QAPP	Bay West	August 2012	September 2012	UFP-QAPP to JBA, USACE, USEPA, and MDE	October 20, 2012
Final UFP-QAPP	Bay West	October 2012	December 2012	UFP-QAPP to JBA, USACE, USEPA, and MDE	December 6, 2012
Field Activities	Bay West	December 2012	December 2012	Analytical data (ongoing), field notes, GPS locations	December 31, 2012
Working Draft Phase I RI Report	Bay West	November 2012	January 2013	Phase I RI Report to JBA and USACE	January 26, 2013
Draft Phase I RI Report	Bay West	February 2013	March 2013	Draft Phase I RI Report to JBA, USACE, USEPA, and MDE	March 11, 2013
Draft Final Phase I RI Report	Bay West	May 2013	June 2013	Draft Final Phase I RI Report to JBA, USACE, USEPA, and MDE	June 13, 2013
Final Phase I RI Report	Bay West	July 2013	July 2013	Final Phase I RI Report to JBA, USACE, USEPA, and MDE	July 30, 2013
A copy of the Integrated Master Summary Schedule from the Project Management Plan dated December 2011 for CS-C503 is included in **Appendix B.**					

QAPP Worksheet #17 – Sampling Design and Rationale

The site background, history, and environmental history as well as the Phase I RI objectives have been provided in Worksheet #10. The PQOs which define the type, quantity, and quality of data are provided in Worksheet #11.

Previous sampling results at CS-C503 detected PCBs in the sediment above the USEPA RSLs. However, sampling to delineate the source and extent of the PCB contamination was not conducted. Due to the limited scope of the historical sampling event, additional contaminant analysis and the extent of contamination is included with this phase of work. Soil/sediment and surface water/stormwater samples will be collected to determine if contamination is present surrounding the retention pond and associated drainage area, where designated on **Figure 4**. The target sample matrices for CS-C503 are based on the conceptual site model included in the Decision Logic Flow Diagram on the next page. The rationale for matrix selection is shown in the following table.

Matrix	Targeted For Sampling	Rationale
Soil	Yes	To determine whether soil contamination is a potential human health and/or ecological concern.
Groundwater	No	PCBs are relatively immobile in the environment and due to their strong affinity to organic carbon, generally are insoluble in water. Therefore, groundwater is not anticipated to be impacted by PCBs.
Soil Vapor	No	There are no buildings at the site and therefore no potential exposure via the VI pathway.
Indoor Air	No	There are no buildings at the site and therefore no potential exposure via the VI pathway.
Sediment	Yes	To determine whether sediment contamination within the retention pond is a potential human health and/or ecological concern.
Surface Water/Stormwater	Yes	PCBs were previously detected in the sediments and the function of the stormwater pond is to provide a settling basin for sediment-bearing stormwater. In addition, PCBs are relatively immobile in the environment and due to its strong affinity to organic carbon, generally is insoluble in water. Surface water is not anticipated to be impacted by PCBs; however, will be sampled at this time to verify that the exposure pathway is incomplete.

The decision logic provided below details the project tasks for collecting soil/sediment and surface water/stormwater samples around the retention pond and associated drainage area, as well as to determine if Dioxin/Furan analysis is warranted. The samples will be collected in accordance with the sampling methodology on Worksheet #14, and sent to TestAmerica Denver for COPC analysis, except in the case of Dioxins/Furans which will be sent to TestAmerica West Sacramento. Concentration levels of analytes in the samples are expected to be low. In the Phase I RI Report, the analytical results will be compared to the screening criteria detailed in Worksheet #11.

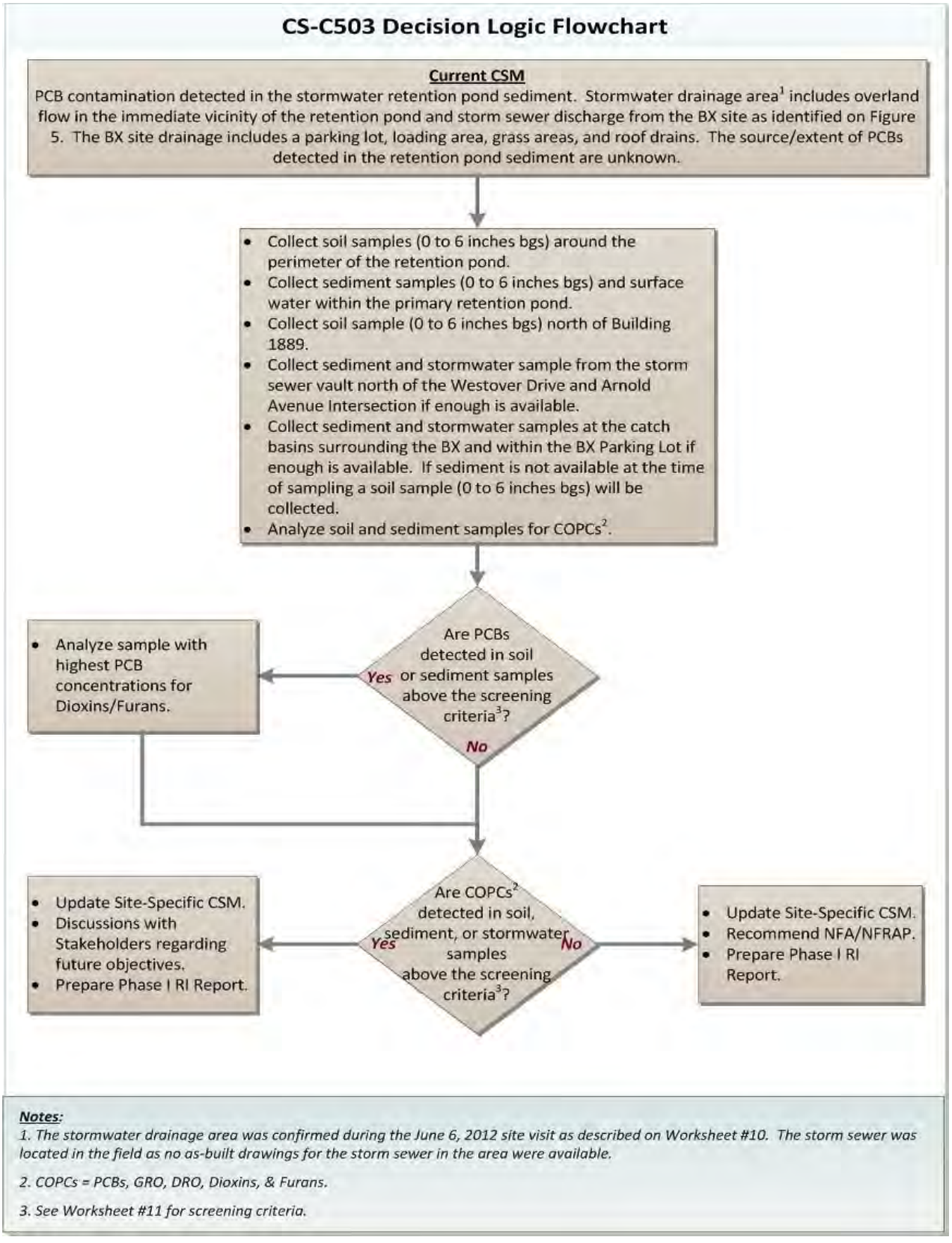
Detailed sampling information may be found in the following Worksheets:

Locations:	Worksheet #18 and **Figure 4**
Location Rationale	Worksheet #18
Matrices:	Soil/Sediment
Analytical Groups:	Worksheet #18

QAPP Worksheet #17 – Sampling Design and Rationale (Continued)

Analytical Method/SOPs:	Worksheets #19, #23, and **Appendix E**
Sampling Frequency:	Worksheet #18
Sampling Schedule:	Worksheet #16
Sampling Methods:	Worksheet #14
# Field Samples:	Worksheet #18
# Field QC Samples:	Worksheet #20

QAPP Worksheet #17 – Sampling Design and Rationale (Continued)



Notes:

1. The stormwater drainage area was confirmed during the June 6, 2012 site visit as described on Worksheet #10. The storm sewer was located in the field as no as-built drawings for the storm sewer in the area were available.

2. COPCs = PCBs, GRO, DRO, Dioxins, & Furans.

3. See Worksheet #11 for screening criteria.

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

Sampling Location ID Number [1]	Matrix	Depth [2] (inches bgs [3])	Analytical Groups [4]	No. of Samples [5]	Sampling SOP Reference	Rationale for Sampling Location
CSC503-01-SB-001	SO	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1	Worksheet #21	Northwest of the northern portion of the retention pond within the drainage area.
CSC503-02-SB-002	SO	0-6		1		On the eastern perimeter in the northern portion of the retention pond.
CSC503-03-SB-003	SO	0-6		1		On the eastern perimeter in the central portion of the retention pond.
CSC503-04-SB-004	SO	0-6		1		On the eastern perimeter in the southern portion of the retention pond.
CSC503-05-SB-005	SO	0-6		1		Adjacent to the perimeter in the southern portion of the retention pond near the inlet apron.
CSC503-06-SW-006	SW	NA	GRO/DRO, PCBs	1		Near the inlet apron in the southern portion of the retention pond.
CSC503-07-SD-007	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Just east of the midline in the southern portion of the retention pond.
CSC503-08-SD-008	SD	0-6		1		Just west of the midline in the southern portion of the retention pond.
CSC503-09-SB-009	SO	0-6		1		West of the southern portion of the retention pond within the drainage area.
CSC503-10-SD-010	SD	0-6		1		Midline in the central portion of the retention pond.
CSC503-11-SD-011	SD	0-6		1		Just west of the midline in the central portion of the retention pond.
CSC503-12-SW-012	SW	NA	GRO/DRO, PCBs	1		Near the midpoint of the retention pond.
CSC503-13-SB-013	SO	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Northwest of the central portion of the retention pond within the drainage area.
CSC503-14-SD-014	SD	0-6		1		West of the midline in the northern portion of the retention pond.
CSC503-15-SW-015	SW	NA	GRO/DRO, PCBs	1		Near the outlet structure in the northern portion of the retention pond.
CSC503-16-SB-016	SO	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		In the woods southwest of the retention pond drainage area.
CSC503-17-SD-017[a]	SD	0-6		1		Within the stormwater vault, north of Westover Drive and Arnold Avenue intersection.

Sampling Location ID Number [1]	Matrix	Depth [2] (inches bgs [3])	Analytical Groups [4]	No. of Samples [5]	Sampling SOP Reference	Rationale for Sampling Location
CSC503-17-ST-018[b]	ST	NA	GRO/DRO, PCBs	1		Within the stormwater vault, north of Westover Drive and Arnold Avenue intersection.
CSC503-18-SD-019[c]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Northwest of the stormwater catch-basin towards the roof drain at the east corner of the BX.
CSC503-18-SB-020[c]	SO	0-6		1		Northwest of the stormwater catch-basin towards the roof drain at the east corner of the BX.
CSC503-18-ST-021[b]	ST	NA	GRO/DRO, PCBs	1		Northwest of the stormwater catch-basin towards the roof drain at the east corner of the BX.
CSC503-19-SD-022[c]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Within the stormwater catch-basin southeast of the BX.
CSC503-19-SB-023[c]	SO	0-6		1		Within the stormwater catch-basin southeast of the BX.
CSC503-19-ST-024[b]	ST	NA	GRO/DRO, PCBs	1		Within the stormwater catch-basin southeast of the BX.
CSC503-20-SD-025[c]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Within the stormwater catch-basin at the BX loading area.
CSC503-20-ST-026[b]	ST	NA	GRO/DRO, PCBs	1		Within the stormwater catch-basin at the BX loading area.
CSC503-20-ST-027[b]	ST	NA		1		Within the stormwater catch-basin at the BX loading area.
CSC503-21-SD-028[c]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Within the stormwater catch-basin southwest of the Burger King parking lot.
CSC503-21-SB-029[c]	SO	0-6		1		Adjacent to the stormwater catch-basin southwest of the Burger King parking lot.
CSC503-21-ST-030[b]	ST	NA	GRO/DRO, PCBs	1		Within the stormwater catch-basin southwest of the Burger King parking lot.
CSC503-22-SD-031[a]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Within the southeasterly stormwater catch-basin in the BX parking lot.

Sampling Location ID Number [1]	Matrix	Depth [2] (inches bgs [3])	Analytical Groups [4]	No. of Samples [5]	Sampling SOP Reference	Rationale for Sampling Location
CSC503-22-ST-032[b]	ST	NA	GRO/DRO, PCBs	1		Within the southeasterly stormwater catch-basin in the BX parking lot.
CSC503-23-SD-033[a]	SD	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Within the easterly stormwater catch-basin in the BX parking lot.
CSC503-23-ST-034[b]	ST	NA	GRO/DRO, PCBs	1		Within the easterly stormwater catch-basin in the BX parking lot.
CSC503-SBXX	SO	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	2		Field Duplicate
CSC503-SDXX	SD	0-6		1		Field Duplicate
CSC503-SWXX	SW	NA	GRO/DRO, PCBs	1		Field Duplicate
CSC503-SBEB01	AQ	0-6	GRO/DRO, PCBs, Dioxins/ Furans, TOC	1		Equipment Blank
CSC503-SBFB01	AQ	0-6		2		Field Blank
CSC503-IDWXX	IDW	NA		TBD		Investigation Derived Waste Samples as needed.

[1]Sample locations are provided in Figure 4.
[2]See Worksheet #14 for sampling rationale.
[3]bgs = below ground surface
[4]Concentrations are expected to be low for all compounds.
[5] Location of field duplicates will be determined based upon field conditions and where concentrations of COPCs are anticipated to be present.
[a] A sediment sample will be collected if enough sediment is present within the designated catch basin (CB) at the time of sampling.
[b] A stormwater sample will be collected if enough stormwater is present at the time of sampling.
[c] A sediment sample will be collected if enough sediment is present within the designated CB at the time of sampling. If sediment is not present in the CB, a soil sample will be collected from the ground surface adjacent to the CB.

SD = Sediment
SB = Soil boring
ST = Stormwater
AQ = Aqueous
IDW = Investigation Derived Waste
SW = Surface Water
EBXX = Equipment/Rinsate Blanks
SBFBXX = Field Blanks

QAPP Worksheet #19 – Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical / Preparation Method SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Soil/Sediment	TPH – GRO	8015C / 5035B, DV-GC-0010	3, 40mL glass Volatile Organic Analysis (VOA) Vials	5.0 grams	2-DI water/frozen and 1-Methanol; ≤6°C but not frozen	48 hours to freeze DI Water 14 days – Preserved 7 days – Unpreserved
Soil/Sediment	TPH - DRO	8015C / 3546, DV-GC-0027 / DV-OP-0015	1, 8oz, glass jar	30 grams	≤6°C but not frozen	14 days to extract – 40 days from extract to analysis
Soil/Sediment	PCB	8082A / 3546, DV-GC-0021 / DV-OP-0015	1, 8oz, glass jar	30 grams	≤6°C but not frozen	14 days to extract – 40 days from extract to analysis
Soil/Sediment	Dioxins	8290A, WS-ID-0005 / WS-IDP-0005	1, 8oz, glass jar[4]	30 grams	≤6°C but not frozen	30 days to extract – 40 days from extract to analysis
Soil/Sediment	TOC	Lloyd Kahn Method	8-ounce glass jar with Teflon lined lid	8 oz	≤6°C but not frozen	28 days
Water	TPH – GRO	8015C / 5030B, DV-GC-0010	3, 40mL glass Volatile Organic Analysis (VOA) Vials	40mL	≤6°C but not frozen; adjust pH <2; HCl 0.008% $Na_2S_2O_3$[4]	14 days – Preserved 7 days - Unpreserved
Water	TPH - DRO	8015C / 3510C, DV-GC-0027 / DV-OP-0006	2, 1 liter, amber	1000mL	≤6°C but not frozen	7 days to extract - 40 days from extract
Water	PCB	8082A / 3510C, DV-GC-0021 / DV-OP-0006	2, 1 liter, amber	1000mL	≤6°C but not frozen	7 Days to extract - 40 days from extract
IDW	As required	As required	1, 8oz, glass jar, or as necessary	30 grams, minimum	≤6°C but not frozen	As required by Analytical Method

[1]Refer to the Analytical SOP References table (Worksheet #23).
[2]The minimum sample size is based on analysis allowing for sufficient sample for reanalysis. Additional volume is needed for the laboratory MS/MSD sample analysis.
[3]Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
[4]Dioxin extraction aliquot will be taken from the PCB sample jar, if needed.

QAPP Worksheet #20 – Field Quality Control Sample Summary Table

Matrix	Analytical Group	Analytical and Preparation SOP Reference	No. of Sampling Locations	No. of Field Duplicates[1]	No. of MS/MSD[2]	No. of Field Blanks[3]	No. of Equip. Blanks[4]	No. of GRO Trip Blanks (one per cooler)	Total No. of Samples to Lab
Soil/Sediment	GRO	DV-GC-0010	24	3	2/2	1	1	1	34
Soil/Sediment	DRO	DV-GC-0027 / DV-OP-0015	24	3	2/2	1	1	0	33
Soil/Sediment	PCBs	DV-GC-0021 / DV-OP-0015	24	3	2/2	1	1	0	33
Soil	Dioxins	WS-ID-0005 / WS-IDP-0005	TBD	TBD	TBD	TBD	TBD	TBD	TBD
Soil	TOC	BR-WC-024	24	3	2/2	1	1	0	33
Water	GRO	DV-GC-0010	10	1	1/1	1	1	0	15
Water	DRO	DV-GC-0027 / DV-OP-0006	10	1	1/1	1	1	0	15
Water	PCBs	DV-OP-0006 / DV-GC-0021	10	1	1/1	1	1	0	15

[1]Collect 1 field duplicate per 10 field samples.
[2]Collect 1 field sample per 20 samples for MS/MSD analysis.
[3]Collect 1 field blank per source of water.
[4]Rinsate for equipment blank is from decontamination of non-disposable equipment.

QAPP Worksheet #21 – Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
SOP-1	Soil Sampling, #016-1510232, 02/10/2012	Bay West	Shovels, trowels, augers, direct push equipment	Yes ☒ No ☐	See Worksheet #14 for additional task requirements
SOP-2	Field Quality Control Samples, #011-1578758, 10/05/2012	Bay West	As described in SOP-1, SOP-9 and the QAPP	Yes ☒ No ☐	See Worksheet #14 for additional task requirements
SOP-3	Field Documentation, #010-1578753, 10/05/2012	Bay West	Daily Diary Forms, Site-Specific logbooks, Permanent marking pen	Yes ☐ No ☒	
SOP-4	Field Equipment Decontamination, #002-1578775, 10/05/2012	Bay West	Decon fluids, buckets, brushes, sprayers, towels	Yes ☒ No ☐	See Worksheet #14 for additional task requirements
SOP-5	Packaging and Shipping of Environmental Samples, #006-1510206, 02/10/2012	Bay West	Coolers, bubble wrap, bags, ice, shipping tape	Yes ☐ No ☒	
SOP-6	Air Monitoring Instrumentation Manual, #007-52523V4, 04/11/2007	Bay West	Monitoring Instrument(s), calibration gases, Tedlar® bags, and maintenance parts.	Yes ☐ No ☒	
SOP-7	Sample Custody, #004-1510208, 02/10/2012	Bay West	CoC Forms, Custody Seals, Logbook	Yes ☐ No ☒	
SOP-8	Classification and Description of Soil, Sediment, and Rock, #007-1577802, 10/02/2012	Bay West	Munsell Soil Color Chart	Yes ☐ No ☒	
SOP-9	Sediment Sampling, #019-1543704, 06/14/2012	Bay West	Ponar® Dredge, Ekman® Bottom Sampler, sample coring device, nylon rope	Yes ☒ No ☐	See Worksheet #14 for additional task requirements
SOP-10	Investigation Derived Waste, #018-129394, 10/02/2012	Bay West	Labels, Manifests	Yes ☒ No ☐	Refer to Worksheet #14 for site-specific IDW procedures.
SOP-11	Groundwater Sampling, #009-1510481, 02/10/2012	Bay West	Pumps, tubing, water level meter, flow-through-cell	Yes ☒ No ☐	See Worksheet #14 for additional task requirements

Notes:
1Any reference to the Site Supervisor in the Bay West SOPs shall mean the FOL for purposes of this UFP-QAPP.
2Refer to **Appendix D** for project sampling field forms.

QAPP Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Photoionization Detector/ Flame Ionization Detector (PID/FID) Air Monitoring Equipment	Calibrate instrument to the Mfg.'s specifications.	Change lamp/clean flame per Mfg.'s specifications.	Instrument specific gases will be used for calibration .	Calibration against known calibration gas values.	Daily or whenever calibration drift is encountered.	± 10% of labeled concentration	Recalibrate with fresh gases, renew/replace sensor(s), change batteries, return to Mfg. for repair.	Site Supervisor	SOP-6

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

QAPP Worksheet #23 – Analytical Laboratory 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?
DV-GC-0027	Diesel and Residual Range Organics (DRO and RRO) by GC/FID (SW-846 Method 8015 and others) Revision 3, 04/04/2012	Definitive	DRO	GC	TestAmerica Denver	Yes ☐ No ☒
DV-GC-0010	Gasoline Range Organics (GRO) by GC/FID SW846 Method 8015 and others) Revision 7.1, 07/29/2011	Definitive	GRO	GC	TestAmerica Denver	Yes ☐ No ☒
DV-GC-0030	Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 Method 8082A) Revision 0.1, 06/11/2010	Definitive	PCBs	GC	TestAmerica Denver	Yes ☐ No ☒
DV-OP-0015	Microwave Extraction of Solid Samples (SW-846 3546) Revision 1, 01/13/2011	Preparation	Organic Prep	N/A	TestAmerica Denver	Yes ☐ No ☒
DV-OP-0006	Revision 7.0, 01/31/2012 Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series	Preparation	Organic Prep	N/A	TestAmerica Denver	Yes ☐ No ☒
WS-ID-0005	Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS, Methods 8290, 8290A & TO-9A Revision 7.3, 12/30/2009	Definitive	Dioxins/Furans	HRGC/HRMS	TestAmerica West Sacramento	Yes ☐ No ☒
WS-IDP-0005	Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS Methods 8290, 8290A & TO-9A Revision 1.1, 02/05/2010	Preparation	Organic Prep	N/A	TestAmerica West Sacramento	Yes ☐ No ☒
BR-WC-024	TOC in Soil; Rev. 0.	Definitive	Soil/TOC	Elemental Analyzer	TestAmerica South Burlington	Yes ☐ No ☒

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?

GC = Gas Chromatograph

HRGC/HRMS = High Resolution Gas Chromatograph/High Resolution Mass Spectrometer

SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #24 – Analytical Instrument Calibration Tables

TCL GRO/DRO Analysis by Method 8015C

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC –8015C GRO & 8015C DRO	Five-point initial calibration for all target analytes	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Relative Standard Deviation (RSD) of CF ≤ 20% Linear – least squares regression r2 ≥ 0.99, r ≥ 0.995	Correct problem then repeat initial calibration	Lab Manager/Analyst	DV-GC-0027, DV-GC-0010
	Initial calibration verification (ICV), must be from a 2nd source.	Immediately following five-point initial calibration	All compounds within 15% of expected value	Correct problem then repeat initial calibration	Lab Manager/Analyst	DV-GC-0027, DV-GC-0010
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All compounds within 15% of expected value and within the RTW.	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.	Lab Manager/Analyst	DV-GC-0027, DV-GC-0010
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW as the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. For questions, see the supervisor or technical director.	Lab Manager/Analyst	DV-GC-0027, DV-GC-0010

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #24 – Analytical Instrument Calibration Tables (Continued)

TCL PCBs Analysis by Method 8082A

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC –8082A	Minimum five-point initial calibration for all target analytes[2]	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	Linear regression correlation coefficient r2 ≥ 0.99, r ≥ 0.995. RSD of CF ≤ 20%	Correct problem then repeat initial calibration.	Lab Manager/Analyst	DV-GC-0021
	ICV must be from a 2nd source	Once immediately following initial calibration	All target compounds within 15% of expected value	Correct problem then repeat initial calibration	Lab Manager/Analyst	DV-GC-0021
	Continuing calibration verification (CCV)	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	All compounds within 15% of expected value and within the RT Window[3].	Correct problem then repeat initial CCV (re-calibrate if necessary) and re-analyze all samples since last successful CCV.	Lab Manager/Analyst	DV-GC-0021
	Retention time window calculated for each analyte (see section 9 for how to calculate RTWs).	System set-up, with each new column or major instrument maintenance. Update the mid-RTW at the start of the run or daily.	Each analyte of the LCS, MS/MSD and CCV must be within the calculated RTW.	Correct the problem and re-process or re-analyze samples. If questions, see the supervisor or technical director.	Lab Manager/Analyst	DV-GC-0021

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
[2]Method 8082A, a five-point calibration is only analyzed for Aroclors 1016 and 1260.
[3]The mean of all calibrated compounds may be used, but all compounds above the 15% must be documented in a NCM
SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #24 – Analytical Instrument Calibration Tables (Continued)

TCL Dioxin/Furan Analysis by Method 8290A

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/HRMS 8290A	Tune / Mass Resolution Check (PFK)	At the beginning and the end of each 12-hour period of analysis.	Resolving power ≥ 10,000 at m/z=304.9842 & m/z=380.9760 + 5ppm of expected mass. Lock-mass ion between lowest and highest masses for each descriptor and level of reference ≤ 10% full-scale deflection.	Retune instrument & verify. Assess data for impact if end resolution is less than 10,000 narrate or reinject as necessary.	Lab Manager / Analyst	WS-ID-0005
HRGC/ HRMS	GC Column Performance Check (CPSM/WDM per method)	Prior to ICAL or calibration verification.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of ≤ 25%; and identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; and absolute retention times for switching from one homologous series to the next ≥ 10 seconds for all components of the mixture.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.	Lab Manager / Analyst	WS-ID-0005
GC/HRMS	Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit. (ICAL)	ICAL prior to sample analysis, as needed by the failure of calibration verification, and when a new lot is used as a standard source for calib verification, internal standard or recovery standard solutions.	RSD ≤ 20% for response factors for 17 unlabeled isomers & 9 labeled IS, and ion abundance ratios within limits specified in SOP; and S/N ≥ 10:1for target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat initial calibration.	Lab Manager / Analyst	WS-ID-0005

QAPP Worksheet #24 – Analytical Instrument Calibration Tables (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	Second-source calibration verification	Immediately following ICAL.	All project analytes within ± 30% of the expected value from the ICAL.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration	Lab Manager / Analyst	WS-ID-0005
	Calibration Verification (CCV)	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios in accordance with SOP; and RF (unlabeled standards) within ± 20%D of average RF from ICAL; and RF (labeled standards) within ± 30%D of average RF from ICAL.	Correct problem, repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV End of Run CCV: If RF (unlabeled standards) > ± 20%D and ≤ ± 25%D and/or RF (labeled standards) > ± 30%D and ≤ ± 35%D of the average RF from ICAL use mean RF from bracketing CCVs to quantitate impacted samples. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labeled), run a new ICAL within 2 hours, and requantitate samples. Otherwise, reanalyze samples with positive detections.	Lab Manager / Analyst	WS-ID-0005

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.
% recovery for each IS in the original sample (prior to dilutions) must be limits in Table per method.
SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

QAPP Worksheet #24 – Analytical Instrument Calibration Tables (Continued)

TOC Analysis by Method 9060A

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Elemental Analyzer	ICAL	Initial Method Setup, after combustion chamber is changed (approximately every 200 drops)	Correlation Coefficient must be >0.995	Perform instrument adjustment and/or maintenance to correct the problem, then repeat ICAL.	TestAmerica Analyst, Department Manager	BR-WC-024
Elemental Analyzer	ICV/CCV	Every 20 drops and at the end of the analytical sequence	%R (85-115)	Perform instrument adjustment and/or maintenance to correct the problem, then repeat ICAL.	TestAmerica Analyst, Department Manager	BR-WC-024
Elemental Analyzer	CCB	After every acetanilide	< QC	Reanalyze samples all samples associated with the CCB if TOC results are < 10 times the CCB concentration.	TestAmerica Analyst, Department Manager	BR-WC-024

ICAL = initial calibration
ICV = initial calibration verification
CCB = continuing calibration blank
CCV = continuing calibration verification
TOC = total organic carbon

QAPP 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	SOP Reference
GC	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	Quality Assurance Manual – Section 20
GC/HRMS	Parameter Setup	Physical check	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TestAmerica Chemist	WS-ID-0005
GC/HRMS	Tune Check	Instrument Performance	Conformance to instrument tuning.	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TestAmerica Chemist	WS-ID-0005
Elemental Analyzer	De-gas pump head when flow is erratic, change analytical columns and bed supports guard, check and replace any damaged or discolored tubing, clean conductivity cell and lubricate left-hand position	QC standards	Check plumbing for leaks, check gases, check pump pressure, check eluant level, and check conductivity meter	Daily, and/or as necessary.	Refer to lab QA manual	Refer to lab QA manual	TestAmerica Analyst, Department Manager	Quality Assurance Manual

QAPP Worksheet #26 – Sample Handling System

Sample Handling System
Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): FOL, and other sampling team personnel/Bay West.
Sample Packaging (Personnel/Organization): FOL or Site Sample Manager /Bay West. Sample temperature is recorded daily prior to shipment and maintained at ≤6°C but not frozen.
Coordination of Shipment (Personnel/Organization): FOL/Bay West. Lab coordinator is called to alert lab of incoming samples. This is especially important for Saturday deliveries.
Type of Shipment/Carrier: Lab courier or overnight shipping.
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Sample Receipt Personnel/ TestAmerica Denver.
Sample Custody and Storage (Personnel/Organization): Sample Receipt Personnel/ TestAmerica Denver.
Sample Preparation (Personnel/Organization): Extractions Personnel/ TestAmerica Denver.
Sample Determinative Analysis (Personnel/Organization): Analytical Personnel/ TestAmerica Denver.
Sample Archiving
Field Sample Storage (No. of days from sample collection): 60 days, or as required on a project-specific basis.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days, or as required on a project-specific basis.
Sample Disposal
Personnel/Organization: Environmental H&S Manager/ TestAmerica Denver.
Number of Days from Analysis: 60 days after the final report is issued.

QAPP Worksheet #27 – Sample Custody Requirements Table

Sample Designation and Tracking System

Samples will be collected in laboratory-supplied sample containers specific to the analysis to be performed. Each sample collected will be assigned a unique sample tracking number used to catalog the results. Each sample will be identified on the sample label by site name, sample location ID, matrix, and a sequential sample ID code as a reference. Sample labels will also indicate the sample matrix (sediment/soil/surface water/stormwater), container type and size, preservative (if any), and analytes. The labels will also have space for the sampler to write the date and time of sample collection and their initials. This information will be entered into the database during sample check-in. A description of the sample (including the sample number, location ID, and sample date and time) will be recorded in the field logbook. Any other pertinent information regarding sample identification will be recorded on the sample log sheets or in the field logbooks. The alpha-numeric coding to be used in the sample system should present the pond location, site name, sample location ID, matrix, and sequential three-digit sample ID code (e.g., CSC503-01-SB-001) as described below.

CSC503	**01**	**SB**	**001**
(Site Name)	(Location ID)	(Matrix)	(Sample ID Code)

Quality Control Samples: Field QC designations will conform to the following formats:

Field Duplicates: Blind field-duplicate samples will be designated in a similar fashion, except that different sequential numbers will be assigned to the duplicate samples. The field logbook will note from which sample location the duplicate was collected. Example: CSC503-01-SB-XXX.

Field Blanks: Field blank sample identifiers will consist of the matrix, an “FB” label, and the sequential number. Example: FBXX.

Equipment/Rinsate Blanks: Field blank sample identifiers will consist of the matrix, an “EB” label, and the sequential number. Example: EBXX.

Investigation Derived Waste Samples: IDW samples will be identified as IDWXX, which will indicate the matrix of the IDW. Example: IDWSS – soil sample, IDWAQ – aqueous sample.

Sample Collection Documentation

A project-specific field logbook will be used to keep daily records of significant events, observations, and measurements during field investigations. The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during field activities. The field logbook also will be used to document all sampling activities. Logbook entries will be made with indelible ink to provide a permanent record, and any errors found in the logbook will be verified, crossed-through, and initialed by the person discovering the error. Field logbooks will be permanently bound and pre-paginated; designated forms will be used whenever possible to ensure that field records are complete. The following items are examples of information that may be included in a field logbook:

- Name, date, and time of entry;
- Names and responsibilities of field crew members;
- Name and titles of any site visitors;

QAPP Worksheet #27 – Sample Custody Requirements Table (Continued)

- Descriptions of field procedures and problems encountered;
- Samples taken at each location;
- Details of sampling location, including sampling coordinates;
- Sample identification numbers of all samples collected;
- Date and time of collection;
- Sample collector;
- Sample collection method;
- Decontamination procedures;
- Field instrument calibration and maintenance;
- Weather conditions;
- Site observations; and
- H&S issues including PPE.

In addition, log of photographs and field sample log sheets will be included as part of the field documentation. A copy of the blank field forms are presented in **Appendix D**.

Field Sample Handling and Chain-of Custody Procedures

All personnel will be instructed about sample custody by the FOL. Custody of samples must be maintained and documented at all times to ensure the integrity of a sample from collection through analysis. Once samples are collected, documentation begins with recording into a field logbook or field sampling form (see **Appendix D**). An accurate written record is necessary to trace the possession and handling of environmental samples. This documentation is referred to as the CoC form. CoC begins when samples are collected in the field and is maintained by storing the samples in secure areas until custody can be passed on. Generally, the FOL or the Site Sample Manager is responsible for insuring proper care and custody of samples are maintained. All samples will be delivered to the laboratory accompanied by a CoC form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

Prior to sample collection, sample containers will be labeled with the sample number, sampler's name, date, and analytical group.

Following collection, samples will be placed on ice in a secure cooler and attended by sampling personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory.

The samples will be shipped to the laboratories in coolers packed with ice and bubble wrap, or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with two signed custody seals to ensure the CoC is maintained. Samples will be shipped to the laboratory by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times for the

QAPP Worksheet #27 – Sample Custody Requirements Table (Continued)

respective analysis is presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

Each sample collected will be assigned a unique sampling tracking number, as described above. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the CoC form. The CoC form is provided by the analytical laboratory and upon completion a copy will be archived in the project files. The following information will be recorded on the CoC form:

- Project name and number;
- Sample matrix;
- Sample collector's name;
- Dates and times of sample collection;
- Sample identification numbers;
- Number and type of containers for each sample aliquot;
- Type of sample preservative;
- QC sample designation;
- Sample preparation and analysis method;
- Special handling instructions;
- Destination of samples;
- Name, date, time, and signature of each individual releasing the shipping container; and,
- Name, address, and individual to receive results.

Laboratory Custody Procedures

Laboratory sample receipt, handling, and custody procedures are provided in more detail in the TestAmerica Denver Quality Assurance Manual. TestAmerica Denver SOP # DV-QA-003 includes laboratory sample management and CoC procedures. At a minimum the following procedures will be included:

- The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the "Temperature Blank" container included in each cooler.
- The samples will be checked according to the TestAmerica Denver "Sample Receiving Checklist" (See laboratory forms included in the Quality Assurance Manual) against the CoC form for holding times, sample identification, and integrity.
- The samples will be logged into the laboratory management system.
- Immediately after receipt, the samples will be stored in an appropriate, secure storage area.

QAPP Worksheet #27 – Sample Custody Requirements Table (Continued)

Custody of the samples will be maintained and recorded in the laboratory from receipt to analysis and this record will be included with the data package deliverables. If the laboratory sample custodian judges sample custody to be invalid (e.g., samples arrive damaged or custody seals have been broken), a “Condition Upon Receipt Anomaly Form” (See form included in **Appendix E**) will be initiated. The Bay West PM will be advised immediately, and the samples will not be analyzed unless the Bay West PM so authorizes. The Bay West PM, the laboratory PM, and QAM will be notified. The Bay West PM will make a decision as to the fate of the sample(s) in question on a case-by-case basis.

The sample(s) will be either processed "as is" with custody failure noted along with the analytical data, or rejected with sampling rescheduled if necessary. Any problem with a sample will be noted in the appropriate data report.

In addition, TestAmerica Denver will follow the laboratory SOP for proper disposal of the environmental samples in accordance with federal, state, and local ordinances.

QAPP Worksheet #28 – Analytical Laboratory QC Samples

Matrix: Soil/Sediment/Water

Analytical Group: GRO/DRO

Analytical Method/ SOP Reference: USEPA 8015C GRO & DRO
DV-GC-0027, DV-GC-0010

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch per matrix	No compounds detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no compounds detected > RL	Evaluate usability of data. If required, reprep/reanalyze Method Blank and all associated samples	Lab Analyst	Accuracy/ Precision	No compounds detected > ½ RL
LCS (LCSD if performed)	One LCS per prep batch per matrix	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.	Evaluate usability of data. If necessary, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available	Lab Analyst	Accuracy/ Precision	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.
MS/MSD	Client specified	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.	Examine the project-specific DQOs. Examine LCS results. Evaluate to assess matrix interference. Contact client for additional corrective action measures if necessary	Lab Analyst	Accuracy/ Precision	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate	All field and QC samples	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.	Correct problem, reprep/reanalyze if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Lab Analyst	Representativeness (Accuracy/Bias)	As specified in Worksheet #12 and the DoD QSM v4.2. If not specified, laboratory's in-house control limits.

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – Analytical Laboratory QC Samples (Continued)

Matrix: Soil/Sediment/Water

Analytical Group: PCBs

Analytical Method/ SOP Reference: USEPA 8082A
DV-GC-0030

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch per matrix	No compounds detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common lab contaminants, no compounds detected > RL	Evaluate usability of data. If required, reprep/reanalyze MB and all associated samples	Lab Analyst	Accuracy/ Precision	No compounds detected > ½ RL.
LCS & LCSD (if performed)	One LCS per prep batch per matrix	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-17, p. G-17.	Evaluate usability of data. If necessary, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available	Lab Analyst	Accuracy/ Precision	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-17, p. G-17.
MS/MSD	Client specified	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-17, p. G-17.	Examine the project-specific DQOs. Examine LCS results. Evaluate to assess matrix interference. Contact client for additional corrective action measures if necessary	Lab Analyst	Accuracy/ Precision	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-17, p. G-17.

QAPP Worksheet #28 – Analytical Laboratory QC Samples (Continued)

Surrogate	All field and QC samples	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-3, p. G-5.	Correct problem, reprep/reanalyze if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Lab Analyst	Representativeness (Accuracy/Bias)	As specified in Worksheet #12 and the DoD QSM v4.2, Table G-3, p. G-5.

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – Analytical Laboratory QC Samples (Continued)

Matrix: Soil/Sediment

Analytical Group: Dioxins

Analytical Method/ SOP Reference: USEPA 8290A
WS-ID-0005

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits[1]	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected ≥ LOD or ≥ 20% of the associated regulatory limit or ≥ 5% of the sample result for the analyte, whichever is greater. (OCDD is considered a common laboratory contaminant and treated accordingly).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Analytical Chemist	Accuracy/Bias Contamination	No target analytes ≥ LOD.
Internal Standard Spike	Every field sample, standard and QC sample	% recovery for each IS in the original sample (prior to dilutions) must be limits in Table per method.	Correct problem, then reprep and reanalyze the samples with failed IS.	Lab Manager / Analyst	Precisions and Accuracy/Bias	Meets all EPA Method requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch	QSM or laboratory statistically derived control limits.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager / Analyst	Accuracy/Bias Contamination	QSM or laboratory statistically derived control limits.
MS/MSD	One MS/MSD per analytical/preparation batch	QSM or laboratory statistically derived control limits, RPD ≤ 20%.	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Lab Manager / Analyst	Precision and Accuracy/Bias	QSM or laboratory statistically derived control limits.

[1]This is a summary of the acceptance criteria; refer to the method SOP for specific or more information.

QAPP Worksheet #28 – Analytical Laboratory QC Samples (Continued)

Matrix: Soil/Sediment/Water

Analytical Group: TOC

Analytical Method/SOP Reference: SM 9060/ DV-WC-0006

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method Blank	One per every batch of 20 or fewer samples	All analytes <1/2 RLs	Re-prep and reanalyze blank and all samples processed with the non-conforming blank.	PM, TestAmerica South Burlington	Accuracy/Bias - Contamination	Detections < RLs
LCS	One per every batch of 20 or fewer samples	QC acceptance control limits listed in TestAmerica SOP DV-GC-0025	Re-prep and reanalyze LCS and all samples processed with the non-conforming LCS.	PM, TestAmerica South Burlington	Accuracy/Bias	%R: 75-125
MS/MSD	Project designated sample matrix QC.	%R: 75-125 %RPD: 20	If result is indicative of matrix interference, discuss in case narrative. Otherwise check for possible source of error, and extract / reanalyze the sample.	PM, TestAmerica South Burlington	Interferences - Accuracy/Bias - Precision	%RPD: 20

QAPP Worksheet #29 – Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes Sample Collection Field Sheet CoC Records Airbills Sample Labels Custody Seals Telephone Logs Corrective Action Forms Digital Photographs	Equipment Calibration Logs, Equipment Maintenance, Testing and Inspection Logs Corrective Action Forms Telephone Logs Daily Quality Control Reports	Sample Receipt, Custody and Tracking Records Standard Traceability Logs Instrument Calibration Logs Sample Preparation Logs Run Logs Equipment Maintenance, Testing and Inspection Logs Non-Conformance Forms or CA Forms Field Sample Results Results for Standards, QC Checks and QC Samples Instrument printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples Manual Integration Summary Data Package Completeness Checklists Sample Disposal Records Telephone logs Electronic and/or hard copies of data reports	Field Sampling Audit Checklists Fixed Laboratory Audit Checklists ERPIMS Submittal Data Validation Reports PT Results (if applicable) QA Results (if applicable) Corrective Action Reports Telephone Logs	UFP-QAPP/WP APP/SSHP CQP Phase I RI Report GIS Source files Sampling Event Tech Memo

QAPP Worksheet #29 – Project Documents and Records Table (Continued)

AFCEE Data Deliverable Requirements

Data deliverables packages requirements have recently changed. Updated information from the AFCEE ERPIMS website is below:

The latest version includes the following software components:

1. ERPToolsX X 5.0 – the latest generation of ERPIMS software developed for distribution to contractors, including prime contractors and laboratories, to assist them in preparing ERPIMS data submissions.
2. ERPIMS Web Service – provides synchronization between the ERPIMS submission and production databases with the client-side database contained within the ERPTools X application.
3. ERPIMS Web Tools – provides data extract and query capabilities to the .mil user community only.

These new tools include automated submission uploads. Customers outside of the .mil domain will be required to obtain an External Certificate Authority (ECA) before they can use the tools. Additional information is accessible from the ERPIMS Registration page.

The name, phone number, and email address of the data submittal point of contact should always be included in the transmittal letter with the data so that an emailed receipt can be provided. Data submissions are subject to additional data validation and should not be considered as a one-time deliverable. After the data is inserted into the AFCEE ERPIMS database, an insert notice will be emailed. The transmittal letter along with items of interest will need to be mailed or shipped to:

HQ AFCEE/OSP, Bldg. 171
2261 Hughes Ave, Ste. 155
Lackland AFB, TX 78236-9853

All courier deliveries (FedEx, UPS, DHL) will use this address:

AFCEE/OSP
3515 S. General McMullen
San Antonio, TX 78226-9853

Data packages will be provided as both hardcopy and portable document format (PDF). Data packages will be Contract Laboratory Program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). The turnaround time for analytical services is 21 calendar days. Turnaround time will be measured from the laboratory receipt of the last samples in a sample delivery group. The sample delivery group must contain no more than 20 samples (and only less if the entire sampling event was comprised of less than 20 samples). Data will be stored by the analytical laboratory for 5 years.

QAPP Worksheet #30 – Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil/Sediment	GRO/DRO	Low	See Worksheet #18	8015C	21 Days	TestAmerica Inc. 4955 Yarrow Street Arvada, CO 80002 Michelle Johnston Tel: 303-736-0110	TestAmerica Inc. 5102 LaRoche Avenue Savannah, GA 31404 Phone 912 354 7858 Bernard Kirkland Tel: 912-354-7858
Stormwater/ Surface Water	GRO/DRO	Low	See Worksheet #18	8015C	21 Days		
Soil/Sediment	PCBs	Low	See Worksheet #18	8082A	21 Days		
Stormwater/ Surface Water	PCBs	Low	See Worksheet #18	8082A	21 Days		
Soil/Sediment	Dioxins	Low	See Worksheet #18	8290A	21 Days	TestAmerica Inc. 880 Riverside Parkway West Sacramento, CA 95605 Bryanna Vandenberg Tel: 916-373-5600	TestAmerica Inc. 4955 Yarrow Street Arvada, CO 80002 Michelle Johnston Tel: 303-736-0110
Soil/Sediment	TOC	Low	See Worksheet #18	9060A	21 Days	TestAmerica Inc. 30 Community Drive, Suite 11 South Burlington, VT 05403 Cathy Kelly Tel. 802-660-1990	TestAmerica Inc. 5815 Middlebrook Pike Knoxville, TN 37921 Jamie McKinney Tel. 865-291-3000

QAPP Worksheet #31 – Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of Corrective Action (title and organizational affiliation)
Field Supervision	Daily during sampling events	Internal	Bay West	Rob Heimbach, Bay West Site Lead, Shirley McMaster, Bay West PM	Jim Hubbell, Bay West FOL/SSHO	Jim Hubbell, Bay West FOL/SSHO	Rob Heimbach, Bay West Site Lead, Shirley McMaster, Bay West PM
Project Supervision	Every Delivery Order	Internal	Bay West	Brad Kulberg, Bay West CQC Systems Manager	Shirley McMaster, Bay West PM	Shirley McMaster, Bay West PM	Brad Kulberg, Bay West Project CQC Systems Manager
Field Sampling System Audit	Every 12 months	Internal	Bay West	John D. Verban, Bay West QAM	Jim Hubbell, Bay West FOL/SSHO	Jim Hubbell, Bay West FOL/SSHO	Brad Kulberg, Bay West Project CQC Systems Manager
Laboratory System Audit	Every 18 months	External	Independent quality assurance contractor	AFCEE's independent quality assurance contractor	AFCEE's independent quality assurance contractor	AFCEE's independent quality assurance contractor	AFCEE's independent quality assurance contractor, Air Force QA Officer Shirley McMaster, Bay West PM

QAPP Worksheet #31 – Planned Project Assessments Table (Continued)

System audits will be performed as appropriate to ensure that the work is being implemented in accordance with the approved project-specific UFP-QAPP and in an overall satisfactory manner.

- The Bay West FOL will supervise the field operations. The FOL will perform a daily check to ensure that the field instruments are calibrated, equipment is properly decontaminated, samples are collected and handled properly, and the fieldwork is accurately and neatly documented. CAs will be implemented immediately if any non-compliance is detected.
- System audits for the laboratory will be performed regularly and in accordance with AFCEE guidance and DoD QSM v4.2 (DoD 2010), as provided in the Laboratory Quality Assurance Manual.
- The Bay West QAM will review the data to ensure that the analytical results were obtained through the approved methodology, and that the appropriate levels of QC were followed. The data review effort will be supervised by the Sampling Contactor QAM.

The Bay West PM will oversee the FOL and QAM, and check that management of the acquired data proceeds in an organized and expeditious manner.

An independent performance audit of field activities may be conducted at the discretion of and under the direction of the JBA or USAF QA officer. If a formal field audit is conducted, the QA officer will check that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved project planning documents and SOPs. These audits and laboratory systems audits will identify the following:

- The assessed entity (e.g., field crew, office personnel, etc. and the associated project, field event, office, etc.);
- Whether the audit is internal or external;
- Location and date(s) of assessment;
- Assessment team members;
- Type of assessment;
- Scope of assessment;
- Documents to be reviewed;
- Notification dates;
- Proposed assessment schedule;
- Assessment number; and,
- Contract number.

Performance audits of laboratories are coordinated through AFCEE and are conducted every 18 months by AFCEE's independent quality assurance contractor. Assessment findings that require CA initiate a sequence of events that include documentation of deficiencies, notification of findings, request for CA, implementation of CA, and follow-up assessment of the CA effectiveness. The procedures for handling any UFP-QAPP deviations and project deficiencies that are identified through the planned project assessments are summarized in Worksheet #32.

QAPP Worksheet #31 – Planned Project Assessments Table (Continued)

Potential problems may involve nonconformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying a condition adverse to project quality will notify the Bay West PM. The PM, with the assistance of the Project CQC Systems Manager, will be responsible for developing and initiating appropriate CA. If the identified deficiencies involve field work, this will be done through the FOL; if the deficiencies involve the laboratory, this will be done through the laboratory PM. The CAs will require follow through to the point of verifying that the CA has been effective. CAs may include re-sampling and/or reanalyzing samples or amending or adjusting project procedures. If warranted by the severity of the problem (for example, if a change in the approved plan is required), the USAF will be notified in writing and the USAF's approval will be obtained before any change is implemented. Minor changes will be documented for the main file by the Sampling Contactor PM. Additional work that depends on a nonconforming activity will not be performed until the problem has been eliminated. The overall CA responsibility for system audits will reside with the Bay West PM. The overall CA responsibility for field audits will reside with the Bay West Project CQC Systems Manager.

QAPP Worksheet #32 – Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Chemistry Field Sampling Technical Systems Audit	Written Field Audit	Shirley McMaster, PM, Bay West, Jim Hubbell, FOL/SSHO, Bay West	5 days after audit	Corrective Action Plan	John D. Verban, QAM, Bay West, Shirley McMaster, PM, Bay West	10 days after receiving notification
Fixed Laboratory Technical Systems Audit	Written Audit Report	Shirley McMaster, PM, Bay West, Michelle Johnston, Laboratory PM, TestAmerica, Inc.	5 days after audit	Corrective Action Plan	John D. Verban, QAM, Bay West, Shirley McMaster, PM, Bay West	14 days after receiving notification
DoD Environmental Laboratory Accreditation Program	Written Audit Report	Shirley McMaster, PM, Bay West, John D. Verban, QAM, Bay West	14 days after audit	Corrective Action Plan	John D. Verban, QAM, Bay West, Shirley McMaster, PM, Bay West, DoD Laboratory Validation Coordinator	14 days after receiving notification
Chemistry Data System Audit	Written Audit Report	Shirley McMaster, PM, Bay West, Michelle Johnston, Laboratory PM, TestAmerica, Inc.	5 days after audit	Corrective Action Plan, Re-submission of data	John D. Verban, QAM, Bay West, Shirley McMaster, PM, Bay West	14 days after receiving notification
Chemistry Data Validation Audit	Written Audit Report	Shirley McMaster, PM, Bay West, John D. Verban, QAM, Bay West	5 days after audit	Corrective Action Plan, Re-submission of data	Shirley McMaster, PM, Bay West	14 days after receiving notification

QAPP Worksheet #32 – Assessment Findings and Corrective Action Responses (Continued)

Field Sampling Technical Systems Audit	Written Field Audit	Shirley McMaster, PM, Bay West, Jim Hubbell, FOL/SSHO, Bay West	5 days after audit	Corrective Action Plan, Re-submission of data	Shirley McMaster, PM, Bay West, Jim Hubbell, FOL/SSHO, Bay West	14 days after receiving notification
Project Supervision	Written Audit Report	Brad Kulberg, Project CQC Systems Manager, Bay West, Shirley McMaster, PM, Bay West	14 days after audit	Corrective Action Plan, Re-submission of data	Shirley McMaster, PM, Bay West, Marty Wangensteen, Program Manager, Bay West	14 days after receiving notification

QAPP Worksheet #33 – QA Management Reports Table

Type of Report	Frequency (e.g., daily, weekly, monthly, quarterly, annually)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per sample delivery group	Within 3 weeks	John D. Verban, Bay West QAM	Shirley McMaster, Bay West PM Molly Maxwell, USACE Project Chemist Lydia Plotz, JBA PM
Major analysis problem identification (internal memorandum)	When persistent analysis problems are detected	Immediately	Brad Kulberg, Bay West Project CQC Systems Manager	Shirley McMaster, Bay West PM
Project monthly progress report[1]	Monthly for duration of the project	Monthly	Shirley McMaster, Bay West PM	Lydia Plotz, JBA PM
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling is occurring	Jim Hubbell, Bay West FOL/SSHO	Shirley McMaster, Bay West PM and/or Lydia Plotz, JBA PM
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately	TestAmerica Denver Laboratory QC Director	Brad Kulberg, Bay West Project CQC Systems Manager John D. Verban Bay West, QAM Molly Maxwell, USACE Project Chemist Lydia Plotz, JBA PM

[1]The monthly progress report is an update for the JBA PM and contract office. The report includes information such as activities completed, an updated schedule, identification of outstanding issues, plans for the next period, and a financial narrative.

QAPP Worksheet #34 – Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Chain of custody	Field personnel are responsible to review and verify chain-of-custody content against the samples within the associated cooler. The original CoC will be taped inside of the lid of the container for shipment. A copy of the chain of custody will be filed with other project documents in the assigned project file.	Internal	Field Sampling Personnel, Bay West
Field notes/logbook	Field sampling data, i.e., field logbooks and field forms, will be maintained. FOL will sequentially number the project logbooks and will review and verify all field generated data. Field forms containing information pertaining to sample collection (i.e., calibration forms) will be forwarded to the PM and placed in the project file.	Internal	Field Sampling Personnel, Bay West Brad Kulberg, Bay West
Audit reports	Upon report completion, a copy of all audit reports will be placed in the project file. If CAs are required, a copy of the documented CA taken will be attached to the appropriate audit report in the project file.	Internal	Shirley McMaster, Bay West Brad Kulberg, Bay West
Analytical data package	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified externally and internally according to the data validation process specified in Worksheet #36 of this UFP-QAPP.	Internal/ External	Brad Kulberg, Bay West John D. Verban, Bay West Michelle Johnston, TestAmerica Denver
Electronic data deliverables	All electronic data deliverables will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received electronic data deliverables will be verified externally and internally against the hard copy laboratory data packages	Internal/ External	Brad Kulberg, Bay West John D. Verban, Bay West Michelle Johnston, TestAmerica Denver Cathy Kelly, TestAmerica South Burlington

QAPP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Data results	Results in summary forms will be checked against the raw data. Calculations will be checked.	Michelle Johnston, TestAmerica Denver Cathy Kelly, TestAmerica South Burlington
IIa	Chain-of-custody forms	Check the sample collection information in the chain-of-custody forms against the sample collection log sheets. The cooler temperature recorded by the laboratory at sample receipt and the pH of the chemical preserved samples will be checked to verify sample integrity from collection to analysis.	John D. Verban, Bay West
IIa	Field logs/sample collection coordinates	Verify that the sampling plan was implemented and carried out as written and any deviation is documented.	Brad Kulberg, Bay West
IIb	Electronic data	Verify that all data have been transferred correctly and completely to the final project data base.	John D. Verban, Bay West
IIb	Laboratory data packages	Ensure that the method-specific QC criteria were met for all samples and analyses. The method criteria or QC criteria generated by the laboratory for the SW-846 methods, or the U.S. EPA Data Validation Guidelines QC limits, will be applied.	John D. Verban, Bay West
IIb	Analytical data deviations	Determine the impact of deviation from the analytical method requirements and matrix interference's effect on the analytical results.	John D. Verban, Bay West
IIb	Project quantitation limits	Verify that the PQLs listed in Worksheet #15 were achieved.	John D. Verban, Bay West

QAPP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa	Soil/Sediment	GRO/DRO, PCBs, and Dioxins/Furans	Low	DoD QSM v4.2, USEPA National Functional Guidelines.	Bay West QAM, Project Chemist, Bay West Data Validation Team
IIa	Stormwater/ Surface Water	GRO/DRO and PCBs	Low	DoD QSM v4.2, USEPA National Functional Guidelines.	Bay West QAM, Project Chemist, Bay West Data Validation Team
IIb	Soil/Sediment	GRO/DRO, PCBs, and Dioxins/Furans	Low	UFP-QAPP Worksheets #s 12, 15, 24, 28.	Bay West QAM, Bay West Data Validation Team
IIb	Stormwater/ Surface Water	GRO/DRO and PCBs	Low	UFP-QAPP Worksheets #s 12, 15, 24, 28.	Bay West QAM, Bay West Data Validation Team

QAPP Worksheet #37 – Usability Assessment

The usability assessment process is used to evaluate and document the usability (i.e., PARCCS) of the data by considering the project DQOs, and whether the data are suitable during the decision-making process. The analytical laboratory will be responsible for reviewing all analytical data generated under this contract to ensure that it meets the requirements of this UFP-QAPP. Each analyst reviews the quality of their work based on established protocols specified in laboratory SOPs, analytical method protocol, project-specific requirements and DQOs.

The intent of the data quality assessment process is to establish the levels of PARCCS and the usability of the final results with respect to the project DQOs. Upon completion of data validation, each data point will be assessed as nonqualified, qualified, or rejected based upon the acceptance criteria, and data validation flags will be added to the project database. These parameters will be based upon the analytical data quality and will encompass the QC criteria established in this UFP-QAPP. Qualification will be given according to the sample delivery group, and will be based on the guidelines as presented in the National Functional Guidelines for Data Review – Dioxins/Furans, and Organics (USEPA, 2011, and 2008b; respectively). Both analytical completeness and contract compliance completeness levels will then be determined for each analytical parameter. Finally, the overall usefulness of the data will be established as related to the project DQOs.

The usability assessment process will consist of reviewing the analytical data validation reports for both usable analytical data (i.e., no validation qualifications or estimated "J"/"UJ" qualifications) and rejected ("R" qualified) analytical data, as well as evaluating the field and analytical data for discrepancies or deviations. This assessment will evaluate the impact of the discrepancies or deviations on the usability of the data and assess whether all the necessary information has been provided for the use in the decision making process. The assessment will assess whether there were deviations in sampling activities (e.g., incorrect sample location or analysis performed), chain-of-custody documentation, or holding times; compromised samples (i.e., damaged samples) and the need to resample; or changes to SOPs or methods that could potentially impact data quality. An evaluation of QC sample results will be performed to assess whether unacceptable QC results (e.g., blank contamination) affect data usability. Data use limitations will be discussed in the quality control reports for data that do not meet the DQOs or DQIs. Other parameters to be evaluated during the usability assessment may include, but are not limited to, the following:

- Matrix effects – matrix conditions (e.g., salt water) that may affect the performance of the extraction or analytical method.
- Site conditions – unusual weather conditions or site conditions that may affect the sampling plan.
- Identifying critical and noncritical samples or target analytes.
- Background or historical data.
- Data restrictions – data that do not meet the project DQOs might be restricted but usable, as qualitative values for limited decision-making purposes.

Precision

Precision is the measure of variability between individual sample measurements under prescribed conditions. Analytical precision is the measurement of the variability associated with

QAPP Worksheet #37 – Usability Assessment (Continued)

duplicate or replicate analyses. Field duplicate, laboratory duplicate, MSD, and LCSD (if analyzed) samples will be used to assess field and analytical precision. The precision measurement will be determined using the Relative Percent Difference (RPD) between the duplicate sample results as follows:

$$RPD = \frac{|A - B|}{(A + B)/2} \quad x \quad 100$$

Where:
A = First duplicate concentration
B = Second duplicate concentration

The RPD limits for precision are presented in Worksheet #28. Associated samples that do not meet the criteria will be evaluated by the Bay West QAM as described in Worksheet #35.

Accuracy

Accuracy is defined as the nearness of a result, or the mean of a set of results, to the true or accepted value. Analytical accuracy is measured by comparing the percent recovery (%R) of analytes spiked into a sample against a control limit. Accuracy will be measured using spiked samples, such as MS, MSD, LCS, and surrogates, if applicable. Surrogates, MS, MSD, and LCS analyzed for contaminants will also be used to assess matrix interferences. Calculation of %R is as follows:

$$Percent\ \ Recovery = \frac{S - C}{T} \quad x \quad 100$$

Where:
S = Measured spike sample concentration
C = Sample concentration
T = True or actual concentration of the spike
The laboratory will review the QC samples and surrogate recoveries for each analysis to ensure that the %R lies within the control limits listed in the UFP-QAPP. Otherwise, data will be flagged by the laboratory.

Representativeness

Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. It is a qualitative parameter that depends on proper design of the sampling program.

The representativeness of data will be maintained by the use of established field and laboratory procedures and their consistent application.

Field personnel will be responsible for collecting and handling samples according to the procedures in this UFP-QAPP so that samples are representative of field conditions. Errors in

QAPP Worksheet #37 – Usability Assessment (Continued)

sample collection, packaging, preservation, or CoC procedures may result in samples being judged non-representative and may form a basis for rejecting the data.

Completeness

Completeness is the percentage of measurements made that are judged to be valid compared to the amount that was expected to be obtained under correct, normal conditions. The completeness goal is to generate a sufficient amount of valid data to meet project needs. To be considered complete, the data set must contain all analytical results and data specified for the project. In addition, all data are compared to project requirements to determine whether specifications were met. Completeness is evaluated by comparing the project objectives to the quality and quantity of the data collected to determine if any deficiencies exist. Data validation and data quality assessment will determine which data are valid and which data are rejected or missing.

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 a rejected (R) flag. The requirement of completeness is 90 percent for samples and is determined using the following equation:

$$Completeness\,(\%) = \frac{Number\ of\ Valid\ \ Measurements}{Total\ Number\ of\ \ Measurements} \times 100$$

Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another, whether it was generated by a single laboratory or during inter-laboratory studies. The use of standardized field and analytical procedures ensures comparability of analytical data. Sample collection and handling procedures will adhere to USEPA-approved protocols. Laboratory procedures will follow standard analytical protocols, use standard units, use standardized report formats, follow the calculations as referenced in approved analytical methods, and use a standard statistical approach for QC measurements.

Sensitivity

Sensitivity is the ability of the analytical test method and/or instrumentation to differentiate between detector responses to varying concentrations of the target constituent. Methodology to establish sensitivity for a given analytical method or instrument includes examination of standardized blanks, instrument detection limit studies, and calibration of the QL. The findings of the usability of the data relative to sensitivity will be included in the report, including any limitations on the data set and/or individual analytical results.

The PARCCS measured performance criteria are described in Worksheets #12, 15, and 28. The following steps will be performed:

QAPP Worksheet #37 – Usability Assessment (Continued)

- Evaluate if the project required quantitation limits listed in Worksheet #15 were achieved for non-detected site contaminants. If no detectable results were reported and data are acceptable for the verification and validation steps, then the data are usable.
- If detectable concentrations are reported and the verification and validation steps are acceptable, the data are usable.
- If verification and validation are not acceptable, the data are qualified, estimated (J, UJ) for minor QC deviations that do not affect the data usability, or rejected for major QC deviations affecting data usability. The impact of rejected data will be evaluated and re-sampling may be necessary. The use of estimated data will be discussed in the project report.
- For statistical comparisons and mathematical manipulations, non-detect values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate will be used to represent the concentration at that sample location.
- Statistical tests will be conducted to identify potential outliers. Potential outliers will be removed if a review of the field and laboratory documentation indicates that the results are true outliers.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
After completion of the data validation and review of the DQI, the data will be reconciled with the measured performance criteria to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and statistical analyses will be performed to estimate the set characteristics. The statistic evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with raised results. The data will be presented in a tabular format. These inspections and statistical analyses will be designed to:

- Identify deviations from the field sampling SOPs
- Identify deviations from laboratory analytical methods
- Identify deviations from this UFP-QAPP
- Identify deviations from the validation process
- Evaluate effects of the above-listed deviations from planned procedures on the quality of the data to meet project objectives
- Identify elevated sample quantitation limits and explain the impact of these results on project objectives
- Identify unusable, rejected data
- Evaluate project assumptions
- Identify unanticipated data-set characteristics such as laboratory variance greater than the sampling variance (i.e., ANOVA, t-test) if enough data are available

QAPP Worksheet #37 – Usability Assessment (Continued)

- Identify and evaluate potential data outliers (95 percent confidence goodness-of-fit test on probability plot data); the plotted data will be transformed, if necessary, depending on the observed distribution
- Evaluate adherence to investigation objectives and decision rules
- Ensure completion of any CAs
- Identify any data gaps

If noncompliant data is rejected, a determination whether resampling will be required will be made in consultation with the USACE project chemist as necessary to meet the performance objectives. The reporting period will be negotiated during consultation with the USACE project chemist. Resampling will be at the contractor's expense.

Identify the personnel responsible for performing the usability assessment:
Bay West QAM, or designee

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
The data will be presented in tabular format; data qualifications such as estimation (J, UJ) or rejection (R) will be applied. Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling if necessary to fill out the data gaps.

References

Air Force Center for Environmental Excellence (Technical Services Quality Assurance Program). 2006. *Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan (QAPP)*, *Final Version 4.0.02*. May.

CH2M Hill. 2004. Basewide Background Study, Andrews Air Force Base, Maryland. March.

CH2M Hill. 2005. Basewide Ecological Risk Assessment Step 7, Andrews Air Force Base, Maryland. March.

Intergovernmental Data Quality Task Force. 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Final.* Version 1. March.

State of Maryland Department of the Environment. 2008. *Cleanup Standards for Soil and Groundwater, Interim Final Guidance, Update No. 2.1.* June.

TolTest, Inc. 2008. *Project Summary Report, Implementation of Best Management Practices for Storm Water Point Source Control – Phase I, Andrews air Force Base, Maryland.* July.

United States Air Force. 11th Wing. 2011. *Joint Base Andrews Environmental Restoration Program Compliance Sites Meeting Minutes.* April.

United States Department of Defense (DoD). 2010. *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 4.2. October.

URS, Inc. 2009. *Volume II Final Evaluation Report, Air Force Compliance Clean-Up Sites, Identification and Evaluation of Defense Environmental Restoration Account (DERA) Eligibility for Air Force Center for Engineering and the Environment (AFCEE), Multiple Locations, Andrews Air Force Base, Maryland.* September.

United States Environmental Protection Agency (USEPA). 2002. *Guidance for Quality Assurance Project Plans*, *(QA/G-5), EPA/240/R-02/009*. December.

USEPA. 2011. *USEPA Analytical Services Branch (ASB) National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review, (OSWER 9240.1-531), EPA 540-R-11-016*. September.

USEPA. 2006a. *Guidance on Systematic Planning using the Data Quality Objectives Process*, *(QA/G-4), EPA/240/B-06/001.* February.

USEPA. 2006b. Reissue Notice May 2006, *EPA Requirements for QA Project Plans*, *(QA/R-5)*, EPA/240/B-01/003. May.

USEPA. 2006c. Region 3 BTAG Screening Benchmarks for Freshwater and Freshwater Sediment. August.

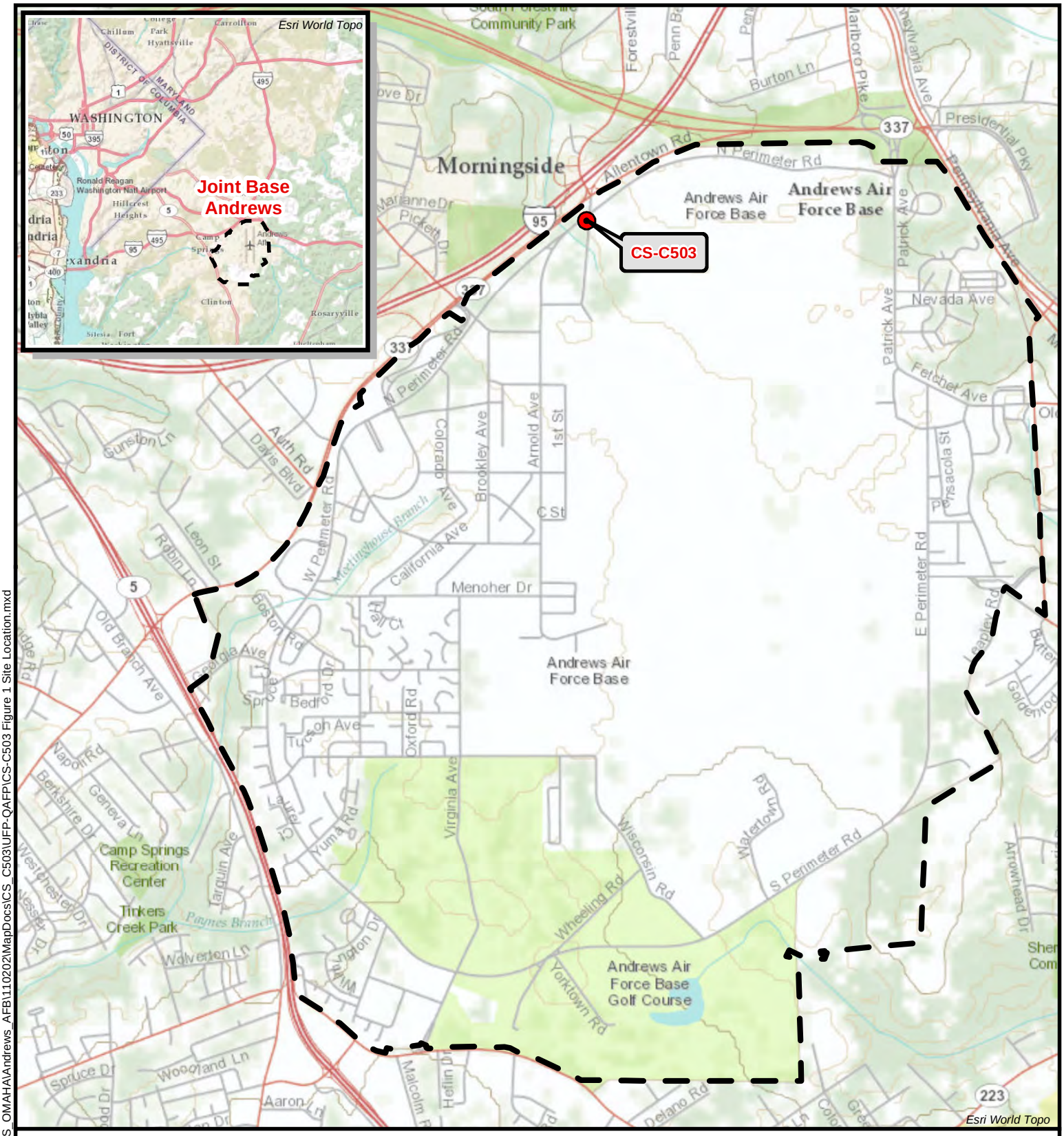
USEPA 2008. Guidance for Developing Ecological Soil Screening Levels (Eco-SSLs). OSWER Directive 9285.7-55. February.

USEPA. 2008a. *U.S. EPA SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. *Third Edition, Update IV.* Office of Solid Waste. January.

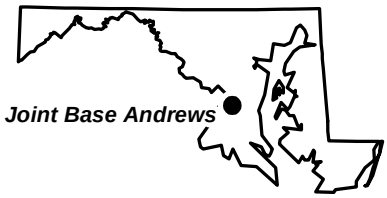
USEPA. 2008b. *Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, (OSWER 9240.1-48), EPA 540-R-08-01*. June.

USEPA/USAF. 2011. Federal Facility Agreement between the Unites States Environmental Protection Agency and the United States Air Force under CERCLA Section 120 in the matter of U.S. Department of the Air Force, Joint Base Andrews, Prince George's County, Maryland. September.

Figures



MARYLAND



N W E S

0 0.25 0.5 1 Miles

0 0.25 0.5 1 Kilometers

Map Projection: NAD_1983_StatePlane_Maryland_FIPS_1900_Feet

Site of Interest

Installation Boundary

Figure 1

CS-C503 Site Location Map

UFP - QAPP

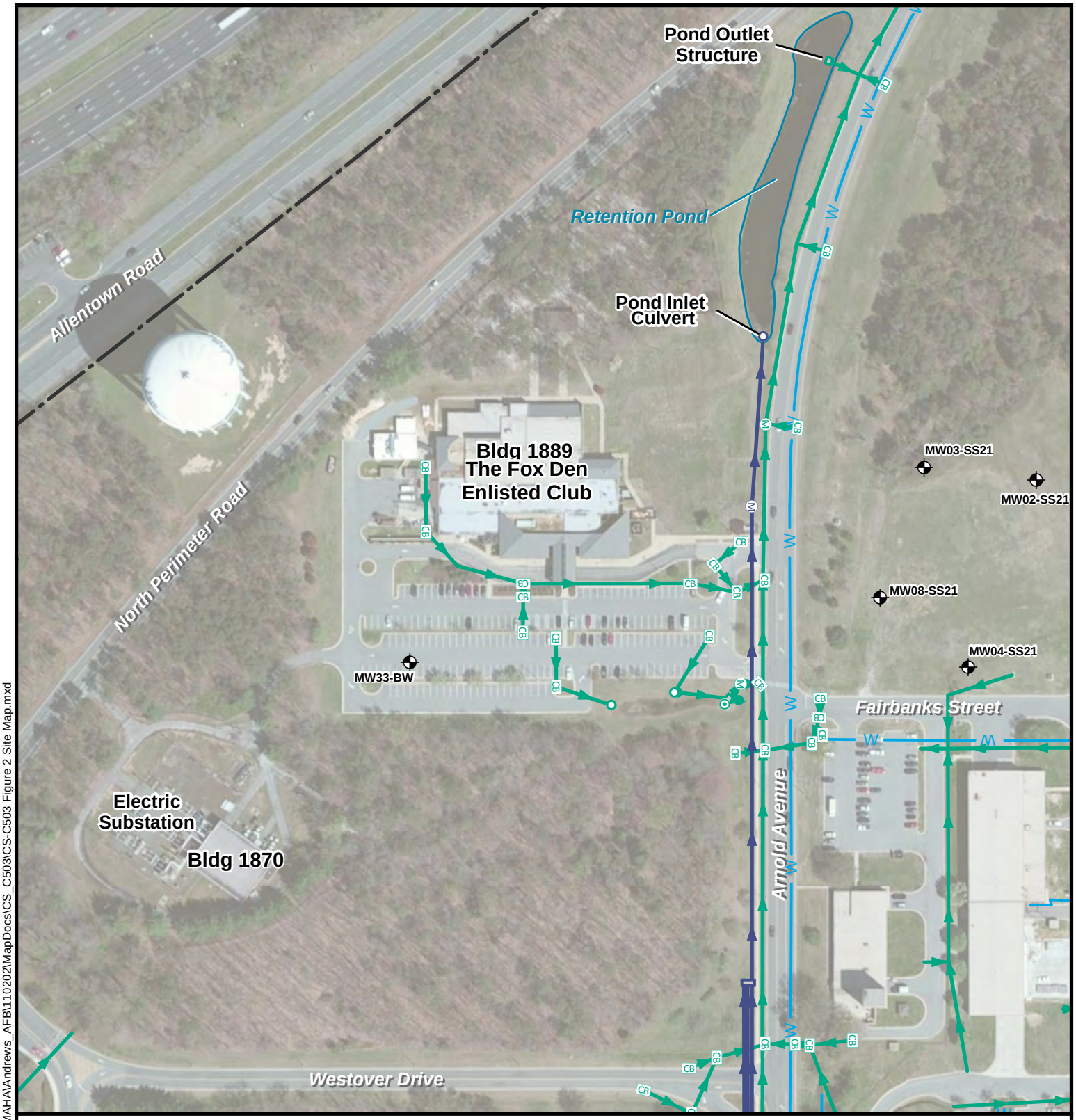
Joint Base Andrews

Camp Springs, Maryland

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: M.B. Date Drawn/Revised: 4/24/2012 Project No. J110202

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\110202\MapDocs\CS_C503\UFP-QAPP\CS-C503 Figure 1 Site Location.mxd



N
W E
S

0 50 100 200 Feet

0 15 30 60 Meters

MARYLAND

Joint Base Andrews

Map Projection: NAD_1983_StatePlane_Maryland_FIPS_1900_Feet

Monitoring Well Location

CB Stormwater Catch-basin

Ⓜ Stormwater Manhole

Stormwater Outflow

W Watermain

CS-C503 Retention Pond Stormwater System

Arnold Avenue Stormwater System

Installation Boundary

Figure 2

CS-C503 Proposed Sample Locations
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: M.B. Date Drawn/Revised: 6/29/2012 Project No. J110202

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\110202\MapDocs\CS_C503\CS-C503 Figure 2 Site Map.mxd

Figure 3A

CS-C503 1964 Aerial Map
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

N W E S

0 100 200 Feet
0 30 60 Meters

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised: 12/18/2012 Project No. J110202

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 3A CS-C503 UFP-QAPP 1964 Aerial Map.mxd

Culvert
Outlet Structure
Inlet Apron
Retention Pond
MW04-SS21
MW03-SS21
MW08-SS21
Jet Engine I/Maintenance Shop Bldg. 1932
SURV Equipment Shop Bldg. 1931
Air Freight Terminal Bldg. 1931
Arnold Avenue
Outlet Structure
Stormwater Vault
Stormwater Vault
3 Parallel Stormwater Sewers
Stormwater Vault
The Club at Andrews Bldg. 1889
Roof Drain
Allentown Road
North Perimeter Road
Westover Dr
MW33-BW
MW05-1845
MW06-1845
MW04-1845
MW08-1845
MW02-1845
MW07-1845
MW03-1845
MW01-1845
Security Police Operations

Existing Features

- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Figure 3B

CS-C503 1974 Aerial Map
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

0 100 200 Feet
0 30 60 Meters

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG | Date Drawn/Revised: 12/18/2012 | Project No. J110202

Jet Engine I/Maintenance Shop Bldg. 1932
Air Freight Terminal Bldg. 1931
SURV Equipment Shop Bldg. 1931
The Club at Andrews Bldg. 1889
Bldg. 1870
Electrical Substation
Security Police Operations
Arnold Avenue
Retention Pond
Culvert
Outlet Structure
Inlet Apron
Outlet Structure
Stormwater Vault
Stormwater Vault
Stormwater Vault
3 Parallel Stormwater Sewers
Roof Drain
Allentown Road
North Perimeter Road
Westover Dr
MW04-SS21
MW03-SS21
MW08-SS21
MW33-BW
MW06-1845
MW05-1845
MW04-1845
MW08-1845
MW02-1845
MW07-1845
MW03-1845
MW01-1845

Existing Features

- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 3B CS-C503 UFP-QAPP 1974 Aerial Map.mxd

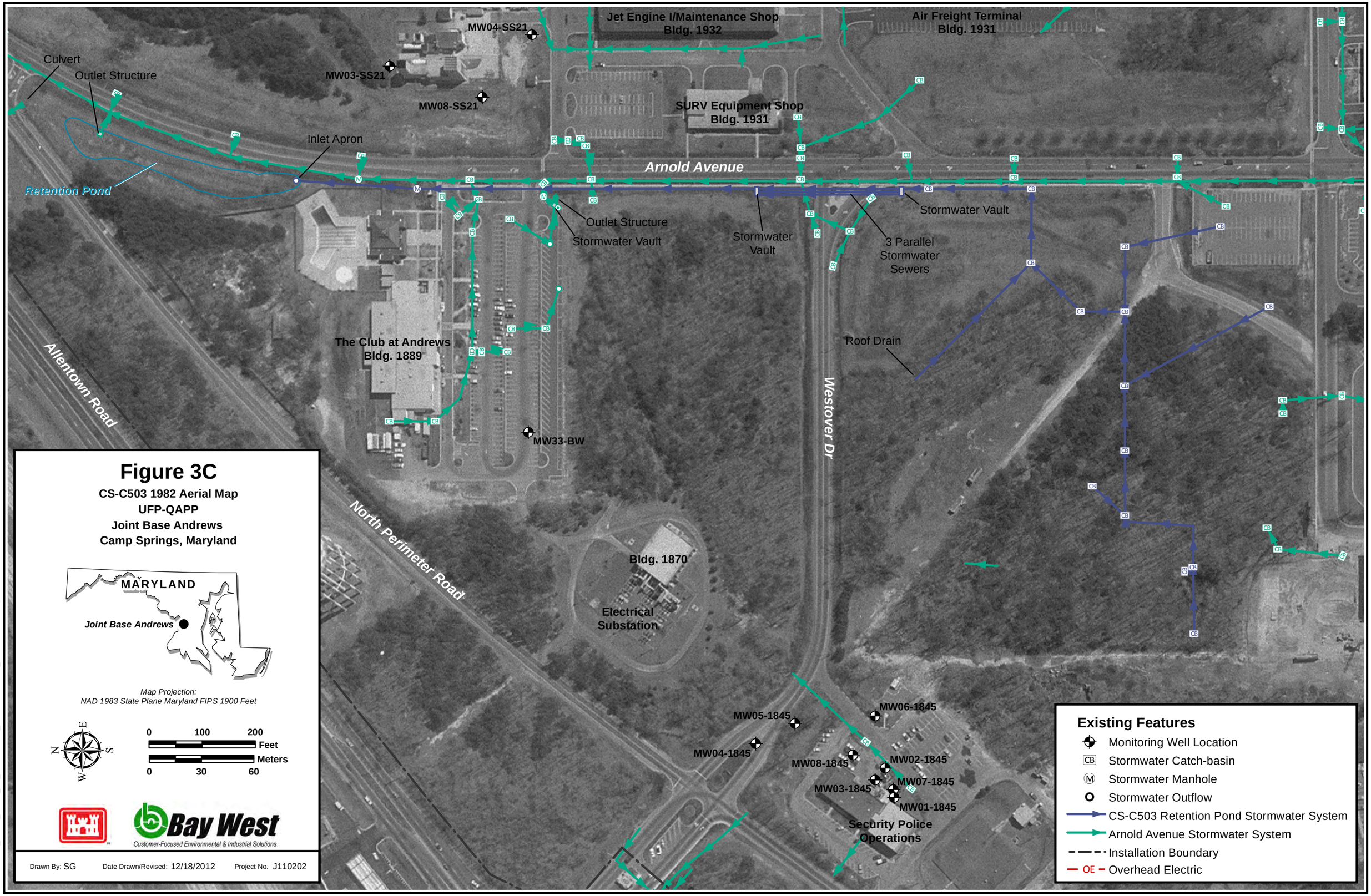


Figure 3C

CS-C503 1982 Aerial Map
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

N W E S

0 100 200 Feet
0 30 60 Meters

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised: 12/18/2012 Project No. J110202

Existing Features

- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 3C CS-C503 UFP-QAPP 1982 Aerial Map.mxd

Figure 3D

CS-C503 2000 Aerial Map
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

N E S W

0 100 200 Feet
0 30 60 Meters

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised: 12/18/2012 Project No. J110202

Jet Engine I/Maintenance Shop
Bldg. 1932

Air Freight Terminal
Bldg. 1931

SURV Equipment Shop
Bldg. 1931

MW04-SS21
MW03-SS21
MW08-SS21
MW33-BW

Culvert
Outlet Structure
Inlet Apron
Retention Pond

Arnold Avenue

Outlet Structure
Stormwater Vault
Stormwater Vault
3 Parallel Stormwater Sewers
Stormwater Vault

The Club at Andrews
Bldg. 1889

Allentown Road
North Perimeter Road
Westover Dr

Roof Drain

Base Exchange
Bldg. 1811

Burger King

Bldg. 1870

Electrical Substation

Brooklyn Avenue

MW05-1845
MW06-1845
MW04-1845
MW08-1845
MW02-1845
MW07-1845
MW03-1845
MW01-1845

Security Police Operations

Existing Features

- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 3D CS-C503 UFP-QAPP 2000 Aerial Map.mxd

Figure 3E

CS-C503 2007 Aerial Map
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

0 100 200 Feet
0 30 60 Meters

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised: 12/18/2012 Project No. J110202

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 3E CS-C503 UFP-QAPP 2007 Aerial Map.mxd

Jet Engine I/Maintenance Shop Bldg. 1932
Air Freight Terminal Bldg. 1931
SURV Equipment Shop Bldg. 1931
MW04-SS21
MW03-SS21
MW08-SS21
Culvert
Outlet Structure
Inlet Apron
Retention Pond
Arnold Avenue
Outlet Structure
Stormwater Vault
Stormwater Vault
3 Parallel Stormwater Sewers
Stormwater Vault
The Club at Andrews Bldg. 1889
MW33-BW
Allentown Road
North Perimeter Road
Westover Dr
Roof Drain
Base Exchange Bldg. 1811
G Street
Bldg. 1870
Electrical Substation
Burger King
Brooklyn Avenue
MW05-1845
MW06-1845
MW04-1845
MW08-1845
MW02-1845
MW07-1845
MW03-1845
MW01-1845
Security Police Operations

Existing Features

- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Figure 4

CS-C503 Proposed Sample Locations
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Map Projection:
NAD 1983 State Plane Maryland FIPS 1900 Feet

0 100 200 Feet
0 30 60 Meters

Drawn By: SG Date Drawn/Revised: 12/18/2012 Project No. J110202

Sample Location	Sample ID per Matrix			
	Sediment	Soil	Stormwater	Surface Water
1		CSC503-01-SB-001		
2		CSC503-02-SB-002		
3		CSC503-03-SB-003		
4		CSC503-04-SB-004		
5		CSC503-05-SB-005		
6				CSC503-06-SW-006
7	CSC503-07-SD-007			
8	CSC503-08-SD-008			
9		CSC503-09-SB-009		
10	CSC503-10-SD-010			
11	CSC503-11-SD-011			
12				CSC503-12-SW-012
13		CSC503-13-SB-013		
14	CSC503-14-SD-014			
15				CSC503-15-SW-015
16		CSC503-16-SB-016		
17	CSC503-17-SD-017[a]		CSC503-17-ST-018[b]	
18	CSC503-18-SD-019[c]	CSC503-18-SB-020[c]	CSC503-18-ST-021[b]	
19	CSC503-19-SD-022[c]	CSC503-19-SB-023[c]	CSC503-19-ST-024[b]	
20	CSC503-20-SD-025[c]	CSC503-20-SB-026[c]	CSC503-20-ST-027[b]	
21	CSC503-21-SD-028[c]	CSC503-21-SB-029[c]	CSC503-21-ST-030[b]	
22	CSC503-22-SD-031[a]		CSC503-22-ST-032[b]	
23	CSC503-23-SD-033[a]		CSC503-23-ST-034[b]	

Bay West
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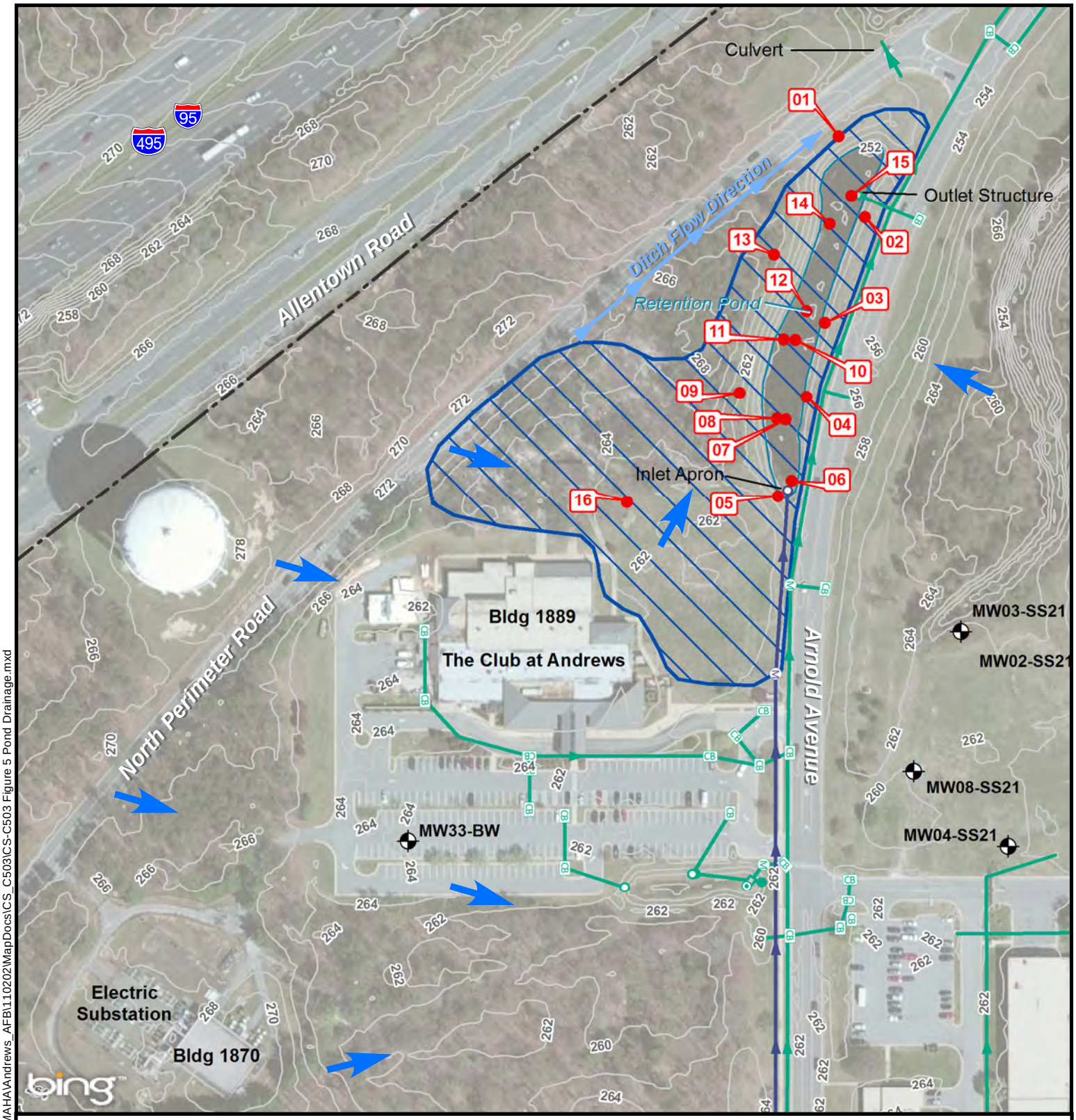
Jet Engine I/Maintenance Shop Bldg. 1932
Air Freight Terminal Bldg. 1931
SURV Equipment Shop Bldg. 1931
Arnold Avenue
Retention Pond
Culvert
Outlet Structure
Inlet Apron
Stormwater Vault
Stormwater Vault
3 Parallel Stormwater Sewers
Roof Drain
Base Exchange Bldg. 1811
The Club at Andrews Bldg. 1889
Bldg. 1870
Electrical Substation
Burger King
Security Police Operations
Allentown Road
North Perimeter Road
Westover Dr
G Street
Brooklyn Avenue
MW04-SS21
MW03-SS21
MW08-SS21
MW33-BW
MW05-1845
MW06-1845
MW04-1845
MW08-1845
MW02-1845
MW03-1845
MW07-1845
MW01-1845

Proposed Investigation
- Sample Location

Existing Features
- Monitoring Well Location
- CB Stormwater Catch-basin
- M Stormwater Manhole
- Stormwater Outflow
- CS-C503 Retention Pond Stormwater System
- Arnold Avenue Stormwater System
- Installation Boundary
- OE Overhead Electric

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\CS-C503\MapDocs\J110202 FIG 4 CS-C503 UFP-QAPP Proposed Sample Locations.mxd



0 85 170 Feet

0 25 50 Meters

N W E S

MARYLAND

Joint Base Andrews

Map Projection: NAD_1983_StatePlane_Maryland_FIPS_1900_Feet

- Soil Sample Location
- Monitoring Well Location
- Stormwater Catch-basin
- Stormwater Manhole
- Stormwater Outflow
- CS-C503 Stormwater System
- Arnold Ave Stormwater System
- Installation Boundary
- Elevation Contour
- Surface Drainage Flow
- Ditch Flow
- Surface Water Drainage Area

Figure 5

CS-C503 Retention Pond
Surface Drainage
UFP-QAPP
Joint Base Andrews
Camp Springs, Maryland

Bay West
Customer-Focused Environmental & Industrial Solutions

Drawn By: SG Date Drawn/Revised: 10/16/2012 Project No. J110202

Y:\Clients\US_ARMY_CORP_OF_ENGINEERS_OMAHA\Andrews_AFB\110202\MapDocs\CS_C503\CS-C503 Figure 5 Pond Drainage.mxd

Appendix A

Background Information

PROJECT SUMMARY REPORT

Prepared For:

Implementation of Best Management Practices for
Storm Water Point Source Control – Phase I
Andrews Air Force Base, Maryland
Project Number: AJXF051769
Contract Number:FA8903-04-D-8678
Task Order Number: 0097

TolTest Project No. 20881

Prepared By:

1480 Ford Street
Maumee, Ohio 43537

2.0 PROJECT ACTIVITIES

To address the performance of the project, TolTest developed an approach to safely and effectively perform the TO in accordance with the Statement of Work (SOW) and approved design package. A summary of the work to repair the Maryland Gate storm water detention pond is described below.

2.1 Summary of Work

The original scope of work included repairing and restoring the Maryland Gate storm water detention pond and its creek bed outfall through the removal and disposal of excess sediment and vegetation found within these storm water control systems/structures. In addition, the creek outfall undercut concrete channel was to be removed and the area repaired or restored in accordance with the Andrews AFB storm water permits, MDE regulations, and the recommended and approved corrective actions and/or restoration structures.

As discussed previously, the project was divided into two distinct phases Phase 1- Maryland Gate Storm Water Detention Pond, and Phase 2-Waterway Stabilization. The MDE approved the design package and project plans for Phase 1. Tasks for Phase 1 were completed and are discussed in the following paragraphs. A signed set of drawings for Phase 2, as well as the delineation maps were completed and delivered to MDE for review and approval. The MDE approved the drawings for Phase 2, however TO funding constraints prevented the completion of the Phase 2 tasks.

The following sections present the full range of project activities that were performed to meet all customer engineering and construction requirements as described in the SOW.

2.2 Regulatory and Permit Issues Permits and Notice of Intent to Construct

The principal organizations involved in permitting during the project were the MDE and Andrews AFB. Additional permits were required by MDE which was a lengthy procedure that impacted the project schedule greatly. TolTest was also responsible for obtaining required clearances from the Base Fire and Rescue and CE departments, such as those required for the temporary disconnection of piping, utilities, and/and hot work permits.

Plans were submitted to the MDE state regulators immediately upon receipt of NTP. Unfortunately, their review of our project was extremely lengthy, and shifted the project schedule significantly prior to our mobilization. The review process and final approval took twenty months to complete for Phase 1.

2.3 Mobilization and Preparatory Work

Prior to the mobilization of personnel or equipment, a pre-construction meeting was held on November 7, 2007 with Andrew AFB personnel including the regulatory agencies to discuss the

project SOW. After the meeting and approval of the project submittals, the procurement of equipment and materials was initiated. Personnel, equipment, and material resources were scheduled to arrive at the project site as they were needed. Mobilization occurred the first week in December 2007.

2.3.1 Site Preparation

The preparatory work involved crew orientation with the project site, setup of an equipment and materials staging/work area, establishment of the laydown area, delineation of the work areas, and the setup of a temporary storm water by-pass for the duration of the construction project. In addition, the perimeter fencing was erected, the construction entrance was installed, and the sump pit was installed. Orientation involved all project personnel, including subcontractors, which included a review of Base security procedures, Base entrance and egress procedures, badging, Base Standard Operating Procedures (SOP) and any specific Base emergency procedures or protocols.

2.4 Site Work

TolTest performed the work activities as specified in the SOW and approved design package. The work activities were in conformance with local and Air Force standards and regulations, as well as all applicable appropriate standards and/or practices. TolTest provided all material, equipment, and labor to complete the following work activities for Phase 1. The following sections provide a summary of the Phase 1 activities. In addition, the monthly Contractor Progress, Status, and Management Reports (CPSMRs) are presented in **Appendix C** for additional reference.

2.4.1 Maryland Gate Detention Pond-Phase 1

TolTest mobilized the subcontractors to Andrews AFB, and immediately began installing the sediment control measures. Silt fencing and a stabilized construction entrance were installed. A temporary dewatering pit was also installed, providing a connection point for dewatering the pond.

TolTest provided for the excavation, removal, and disposal of excess sediment, trees, and vegetation found in the stormwater detention pond. The overall pond dimension was approximately 430 feet long by 45 feet wide. The approximate area of sediment/vegetation removal was 220 feet long by 35 feet wide. The amount of sediment removed from the pond area was approximately 870 tons. The objective was to remove enough sediment and vegetation to sufficiently repair and return the stormwater structure to proper working order. TolTest also located the previously installed outfall pipe, which consisted of a short (~4 foot) section of 4-inch perforated pipe with a tee (presumably a cleanout), and a 90 degree elbow on its end turned down with holes drilled into it, and bedded on stone on the end of the elbow. This piping was to convey pond water into the concrete structure, but appeared to be not functioning properly.

TolTest established an appropriate temporary stormwater bypass system/structure to prevent the detention pond from refilling during site activities in the event of a storm(s). Bypass pumping was utilized in conjunction with a flow-through bladder, and also included a 4-inch dry-prime diaphragm pump and adequate lengths of hose and/or pipe to connect diverted stormwater from the inlet structure to the outlet structure. In addition, one additional 4-inch pump was utilized as daily water removal- it was connected to the dewatering pit that TolTest installed as part of our mobilization.

2.4.1.1 Groundwater Upwelling

TolTest encountered a consistent upwelling of groundwater into the pond as we were excavating the accumulated sediment. Part of the SOW was to clean sediment from the stormwater detention pond to its original elevations. While removing the sediment from the bottom, groundwater seeps were encountered. This changed field condition allowed the bottom of the pond to partially fill with water and continued to fill to static equilibrium with the perched water table.

A dewatering pit was installed to assist with the removal of the water, preventing "water contamination" of the sediment. Visual inspections were conducted of the outflow water during the pumping and the water remained clear throughout the duration of the pumping/ dewatering process. TolTest continued pumping the pond 24-hours a day 7-days a week in order to prevent sediment from going down the outfall and into the stream, which would have been a violation of the State of Maryland surface water regulations. This site condition change required additional labor, equipment rental, fuel charges to be incurred, and maintenance which forced us to revisit/descope the SOW for the stream.

TolTest proposed two options to deal with groundwater inflow to the Base, AFCEE, and MDE regulators to consider-Option 1 a dry bottom pond, or Option 2 a wet bottom pond. Option 1 posed several obstacles and a potential cost growth with trying to establish a dry bottom pond due to significant changes to the structure and elevations of the pond bottom that would be required. Furthermore, an inflow of groundwater complicated the issue since groundwater could not be discharged to the storm water system according to state regulations. Option 2 could be achieved with little/no elevation changes to the pond bottom, no additional material to haul in, and pump maintenance could cease once the pond banks were stabilized. The outfall piping installed would be a relatively simple low-flow device to be approved by MDE, and would likely be installed vertically onto the concrete structure. Aesthetically the pond would resemble preconstruction conditions, with the exception that there would be no vegetation (with the exception of turf grass on the slopes and the remaining trees). It was decided that the Base preferred Option 2. MDE gave their concurrence to install a dewatering device of perforated piping, wrapped in filter fabric and wire mesh, set in a bed of gravel.

2.4.1.2 Stream Inlet and Effluent Structures

The MDE directed that the swale leading to the inlet of the pond needed to be stabilized. In addition, repairs to the effluent structure needed to be performed; specifically the concrete riser was to be repaired by removing the clogged flexible piping, filling in the 4-6" inlet bottom with

concrete or equivalent, and sealing the joints that were leaking between the pre-cast sections of the effluent structure.

TolTest repaired the stormwater concrete inlet that processed effluent water from the stream. TolTest (at the Base's request) installed a new piping system at the existing concrete structure, which comprised of 40 linear feet (LF) of 4-inch perforated schedule 40 pipe, wrapped in ¼-inch galvanized wire, which was covered in filter fabric, and that piping system was bedded, and covered in clean ¾"-1-1/2" blue stone. Specific details of the installed piping are as follows:

From the north corner of the concrete outfall (in the pond), at a 45 degree angle, we installed 20 LF of pipe (wrapped in ¼" x ¼" galvanized "rat wire" wrapped in filter fabric), at the end of the first 20 LF section, and at a 45 degree angle to the first section of pipe, we installed an additional 20 LF of pipe (same configuration as above), and at the end of that section, we installed a 3' vertical riser. All piping was bedded, and covered by ¾"-1-1/2" blue stone, and the piping was installed at a ½" per LF fall back to the concrete structure, giving a total of 2" of fall in the 40 LF.

2.4.1.3 Regulatory Decisions

TolTest worked with the MDE regulators to address the Base's concerns regarding the effluent piping/penetration/water from the pond, as well as potential flooding concerns due to the MDE directive to block the bottom penetration of the concrete structure. MDE response was that based on the information provided to the MDE office (reports-including elevations of influent piping, effluent piping, as well as estimated equilibrium; drawings; and photos), it was their belief that this pond was intended to be a sediment trap supporting the construction of a separate site, and that this was never converted to a permanent stormwater detention pond.

Without original drawings of the pond, and the absence of an "effluent piping system", MDE implied that they could not come to any other conclusion. MDE also indicated that it was the opinion of the office that this pond should have been, and was designed with the 5 and 10 year storms in mind, and that the weir outflow should be sufficient to prevent any flooding.

MDE however indicated that they would review the provided delineation maps, and might concede to the Base's request for an effluent penetration in the effluent structure based on the information contained in those maps, should it sufficiently indicate that a penetration lower in elevation would be necessary.

The Base CE spoke with the MDE on May 6 about the stormwater pond, and that it was originally designed for and presently serves only the Base Exchange and no other drainage area. It was never used as a sediment trap. Base CE also related recollection of the original dewatering device. MDE gave their concurrence to install a dewatering device of perforated piping, wrapped in filter fabric and wire mesh, set in a bed of gravel.

2.4.1.4 Site Restoration

On May 20, the pond work was completed and the MDE approved the stream work. TolTest restored the surrounding impacted areas with Base approved turf grass seed, as well as an appropriate amount of fertilizer, and lime, and covered it with erosion control matting, and ensured that it received appropriate water to germinate the seed. Also completed was the removal of the perimeter fence and trailer, and seeding/fertilizing the lay-down area. The lay-down yard was covered with Curlex to promote rapid germination of the seed.

2.4.2 Detention Pond Outfall/Waterway Stabilization-Phase 2

In light of the Phase 1 field activities, the changed site conditions (dewatering of upwelling groundwater and additional stabilization), and their budgetary impacts, TolTest was unable to perform all of the activities outlined in our original scope of work. AFCEE and the Base agreed to de-scope some of the stream work. The areas which TolTest were not able to provide service to complete were the concrete outfall, the reverse arrow debris trap, and the rip-rap stabilization of the stream bends.

We were however able to remove 11 trees from the stream area adjacent to the wetland (as directed by the Base). TolTest and the Base agreed that the trees posed the most immediate threat to the proper flow of the stream outfall system, and chose to remove them. TolTest removed the trees that were threatening to either fall into the stream, or those whose root masses had been exposed due to years of erosion at the bases of these trees. The larger sections of trees were hauled off base, and disposed of, while the smaller portions were fed into a chipper, and returned to the forest floor and placed over the track marks made by heavy equipment (as directed by the Base).

2.5 Waste Characterization Sampling and Disposal

Prior to removing and disposing of pond sediment/sludge, TolTest collected two composite samples from the detention pond sediment for disposal characterization analysis including toxicity leaching procedure. The samples were collected from the influent and effluent areas of the pond. The samples were analyzed by TestAmerica Laboratories, Inc for volatile organic compounds, semi-volatile organic compounds, PCBs, pesticides, herbicides, metals, mercury, total petroleum hydrocarbons, cyanide, sulfides, pH, and ignitability. Analytical results are presented in **Appendix D**.

Per the characterization analysis, all waste generated was approved by the MDE for non-hazardous disposal provided that the material passed a paint filter test. Attempts were made to dry the sediment by placing it onto the banks of the pond, and draining the water from the material. We also introduced wood chips as necessary to dry the material. Total quantity of soil/sediment that was disposed was approximately 700 cubic yards (870 tons) and disposed as daily cover material at the Brown Station Road Landfill in Prince George's County. The soil acceptance letter is presented in ***Exhibit 3***.

2.6 Demobilization

TolTest scheduled a final inspection to ensure all tasks were completed to the specifications and the satisfaction of the client on June 12, 2008. A punch list of any remaining items was not needed, as the Base, and the AFCEE representative present approved of the corrective measures taken on this TO.

2.6.1 Temporary Facilities

Temporary contractor facilities were removed and demobilized after all site activities were completed and approved by Andrews AFB and AFCEE. All construction debris and temporary facilities, barriers, etc. were removed and the areas returned to their original condition.

2.6.2 Construction Equipment and Facilities

TolTest demobilized all equipment and personnel at project completion. Demobilization was coordinated with site security to ensure a smooth transition off site, without impact to daily operations.

CONTRACTOR PROGRESS, STATUS, AND MANAGEMENT REPORT (CDRL B004)
DECEMBER 1 THROUGH DECEMBER 30, 2007

Contract No. FA8903-04-D-8678, TO 0097

Prepared For:

Department of the Air Force
Air Force Center for Engineering and the Environment
3300 Sidney Brooks Road
Brooks City-Base, TX 78235-5119

TolTest Project No. 20881

1480 Ford Street
Maumee, Ohio 43537

January 2008

TABLE OF CONTENTS

Section **Page No.**

1.0 INTRODUCTION

This Contractor's Progress, Status, and Management Report (CPSMR) is being submitted for the month of December, 2007. TolTest was awarded Task Order (TO) 0097 under the Air Force Center for Engineering and the Environment (AFCEE) Contract (No. FA8903-04-D-8678). The work being performed under TO 0097 is for Implementation of Best Management Practices for Storm Water Point Source Control – Phase I. The TO award was made on March 14, 2006 pursuant to the Statement of Work (SOW) dated February 3, 2006.

Report Preparer:

Robert R. Beckwith
Project Manager
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Phone: 419-794-3570
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E-mail: bob.beckwith@toltest.com

1.1 Purpose and Objectives

The purpose of this TO is to provide all labor, materials and expenses needed to complete the Implementation of Best Management Practices for Storm Water Point Source Control – Phase I .

1.2 CPSMR Organization

Data Item Description DI-MGMT-80227 with TO specific tailoring was utilized as the outline for this submittal. This CPSMR includes:

Section 2.0	Site Progress Reporting
Section 3.0	Project Organization
Section 4.0	Problem Areas
Section 5.0	Significant Items, TO Modifications
Section 6.0	Contract Schedule Status
Section 7.0	Activities Planned for Next Reporting Period

2.0 SITE PROGRESS REPORTING

This report covers the project progress from December 1 through December 30, 2007. The following sections provide a discussion of project progress.

2.1 Project Progress

During the first week of December, TolTest completed the mobilization, and have erected the perimeter fencing, installed the construction entrance, and installed the sump pit. The pond was dewatered, and the composite material samples were taken and were shipped to Severn Trent Laboratories (now Test America) office in Pittsburgh for full waste characterization

While waiting for the analytical results, the subcontractor temporarily demobilized, to return to the site immediately upon receipt of the analytical results. The machinery, and site controls remained in place during this waiting period, and the subcontractor will reinstall the sump pit upon their return, which was removed to remove accumulated silt from filter fabric.

Results were receive on December 18, 2007. Two samples were collected and both contained PCBs (190 and 300 ug/kg) and TPH as diesel (120 and 640 mg/kg). Our assumption was that the material would be disposed as non-hazardous. The selected, nearby landfill, DC Materials has a zero tolerance policy on PCBs. Their policy dictates that if any PCBs are detected in analysis (regardless of concentration), then they will not accept soils. We have searched for other surrounding disposal centers/landfills to see if they will accept this material as non-hazardous.

Also in December, TolTest received a letter from the MDE regarding the Wetlands and Waterways Permit. The MDE Project Manager requested additional information: compute velocities and tractive forces necessary for a 2-year and 10-year frequency storm events; full sized drawings; and flood plain notifications.

A meeting will be conducted on January 3, 2008 to discuss the sediment disposal and permit issues with AAFB and TolTest personnel.

2.2 Project Cost Summary

Project Metrics as of the end of Current Reporting Period			
Project Award		ACRN AA	$332,917
Percent Complete	31%	Current Invoice	$31,463
Percentage Billed	31.24%	Total Invoiced	$104,007

3.0 PROJECT ORGANIZATION

The project organization has not changed from the last reporting period.

4.0 PROBLEM AREAS

Problem areas are related to landfill acceptance of the pond sediment and wetland and waterway permit. We are working with DC Materials landfill to accept the sediment as non-hazardous

wastes, since it is less than the Maryland criteria of 50 ppm. If the material needs to go to another disposal facility, TolTest will need to request a contract modification.

The MDE regulatory review process is taking longer than anticipated. TolTest did not include the funding required to obtain wetland and waterway permit from the MDE in our original proposal. At the original kick off meeting with AAFB personnel and the MDE (attended by Ms. Amanda Malcolm and Mr. Bob Cooper) it was indicated that this project was not large enough to require a wetland and waterway permit. If this permit is required TolTest will need to request a contract modification.

5.0 SIGNIFICANT ITEMS, TO MODIFICATIONS

No significant items or modifications occurred during this period.

6.0 PROJECT SCHEDULE STATUS

TolTest may prepare and submit an additional POP extension request (if necessary) to allow for the extended regulatory review time relating to the wetland and waterway permit.

7.0 ACTIVITIES PLANNED FOR NEXT REPORTING PERIOD

Activities planned for January 2008 include obtaining landfill acceptance of the pond sediment and working on the wetland and waterway permit issues. TolTest will contact its engineering department to determine which information it needs in order to find the appropriate values for the 2-year and 10-year frequency storm events, and TolTest will contact the Prince George County Dept. of Planning to obtain a copy of the FEMA 100-year Floodplain Analysis for the county. AAFB CEVQ will provide TolTest a copy of the floodplain assessment for AAFB.

VOLUME II

FINAL EVALUATION REPORT

ANDREWS AIR FORCE BASE

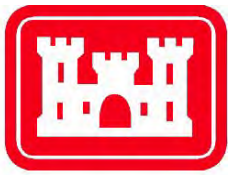
U.S. Environmental Protection Agency
Region 3
Air Force District of Washington

Air Force Compliance Clean-Up Sites, Identification and Evaluation of Defense Environmental Restoration Account (DERA) Eligibility for Air Force Center for Engineering and the Environment (AFCEE), Multiple Locations

Contract No. W912QR-04-D-0025
Task Order DS03

September 2009

Prepared for:
U.S. Army Corps of Engineers Tulsa District and
Air Force Center for Engineering and the Environment



Prepared by:
URS Group, Inc.
12120 Shamrock Plaza, Suite 300
Omaha, Nebraska 68154

URS

3.39.3 Evaluation and Recommendation

The site is located on land owned by the U.S. and under jurisdiction of the Secretary of Defense and was owned by the U.S. and under jurisdiction of the Secretary of Defense at the time of contamination. The sampling analytical results indicate that there may have been a release of fuel from the AST. Additionally, none of the activities conducted at this site were considered ineligible under the criteria discussed in the Background section of this Evaluation Report. Based on these factors, this site is considered DERA(IR) eligible and is recommended for validation. Additional details concerning specific DERA(IR) eligibility criteria evaluated for this site are included in Appendix D.

3.40 PCB-CONTAMINATED SEDIMENT IN RETENTION POND (CS-C503)

3.40.1 Site Description

PCB-contaminated sediment was detected in a recently reconstructed retention pond that is approximately 500 feet by 50 feet in an area located at the intersection of Arnold Avenue and North Perimeter Road. An electrical substation is located within 500 feet of the retention pond.

3.40.2 Sampling and Analysis Activities

No sampling activities were recommended or completed for this site under this project task order.

3.40.3 Evaluation and Recommendation

The site is located on land owned by the U.S. and under jurisdiction of the Secretary of Defense and was owned by the U.S. and under jurisdiction of the Secretary of Defense at the time of contamination. There has been a release of PCBs at the site. Additionally, none of the activities conducted at this site were considered ineligible under the criteria discussed in the Background section in Volume I of this Evaluation Report. Based on these factors, this site is considered DERA(IR) eligible and is recommended for validation. Additional details concerning specific DERA(IR) eligibility criteria evaluated for this site are included in Appendix D.

3.41 UNDERGROUND STORAGE TANK SITE 3117 (UST 3117)

3.41.1 Site Description

This is an UST removal site. There is the potential for soil contamination from the UST; however, no records could be found for the UST, and a Release Assessment is required. The site is listed as MDE OCP Case No. 1995-0350-PG1.

PHOTOGRAPHIC LOG – Andrews Air Force Base, Maryland

Client Name:	Site Location:	Project No.
USACE – Tulsa District; Contract W912QR-04-D-0025, Task Order DS03	CS-C503, PCB-Contaminated Sediment in Retention Pond	16170415

Photo No. 1	Date: 01/13/09

Description:
View of retention pond with Arnold Avenue on the right, facing north

Photo No. 2	Date: 01/13/09

Description:
View of retention pond with Arnold Avenue on the left, facing south

316th Wing

New Sites as listed in the FFA.
Initial Descriptions and Discussions

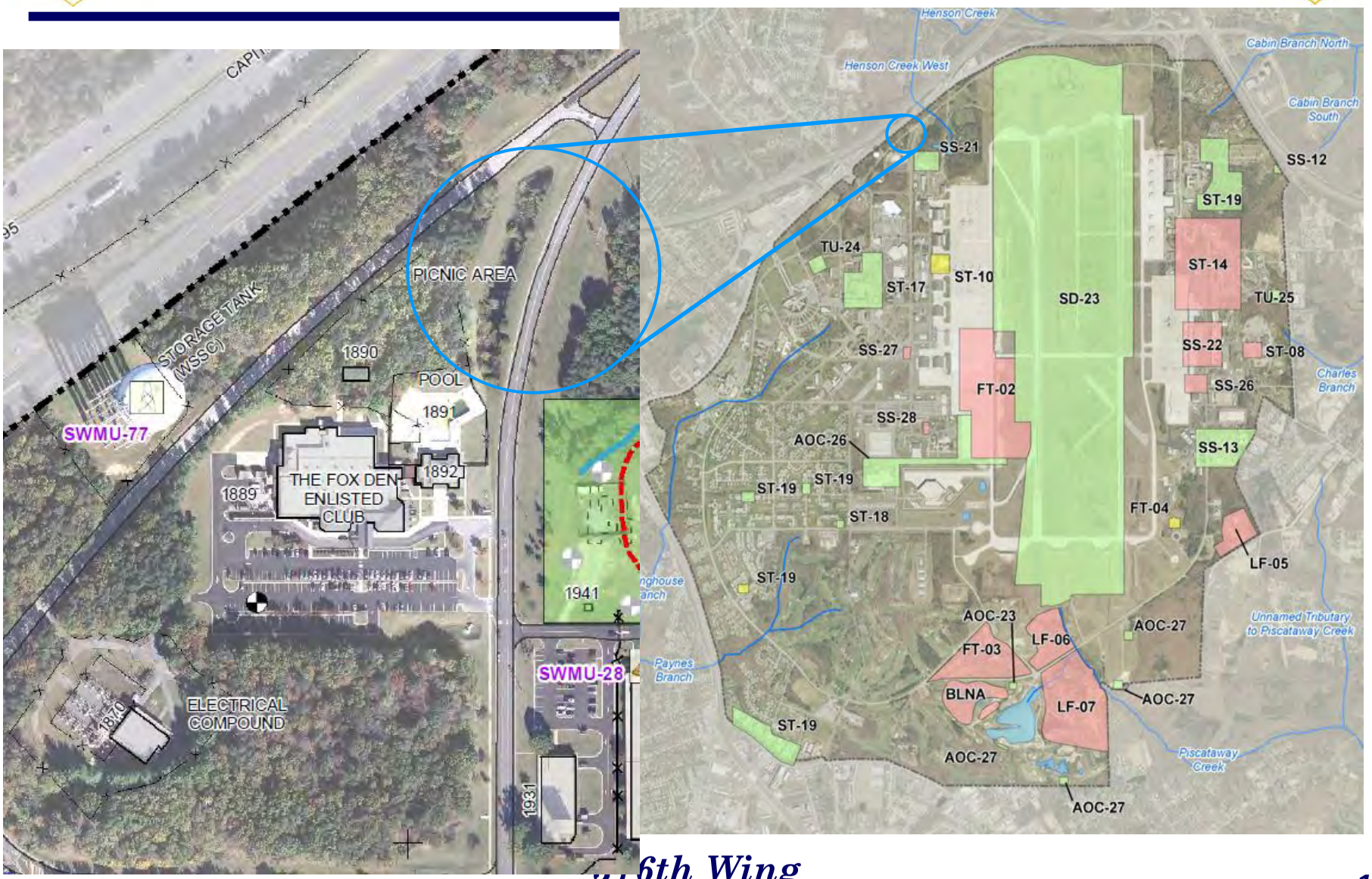
AAFB Tier I Partnering Team Meeting
February 2010

CS-C503

- PCB-contaminated sediment was detected in a recently reconstructed retention pond that is approximately 500 feet by 50 feet in an area located at the intersection of Arnold Avenue and North Perimeter Road.
- An electrical substation is located within 500 feet of the retention pond.

- AROCLOR1254: 300.0 ppb in soil/sediment
- Total Petroleum Hydrocarbons (TPH): 640 ppb in soil/sediment

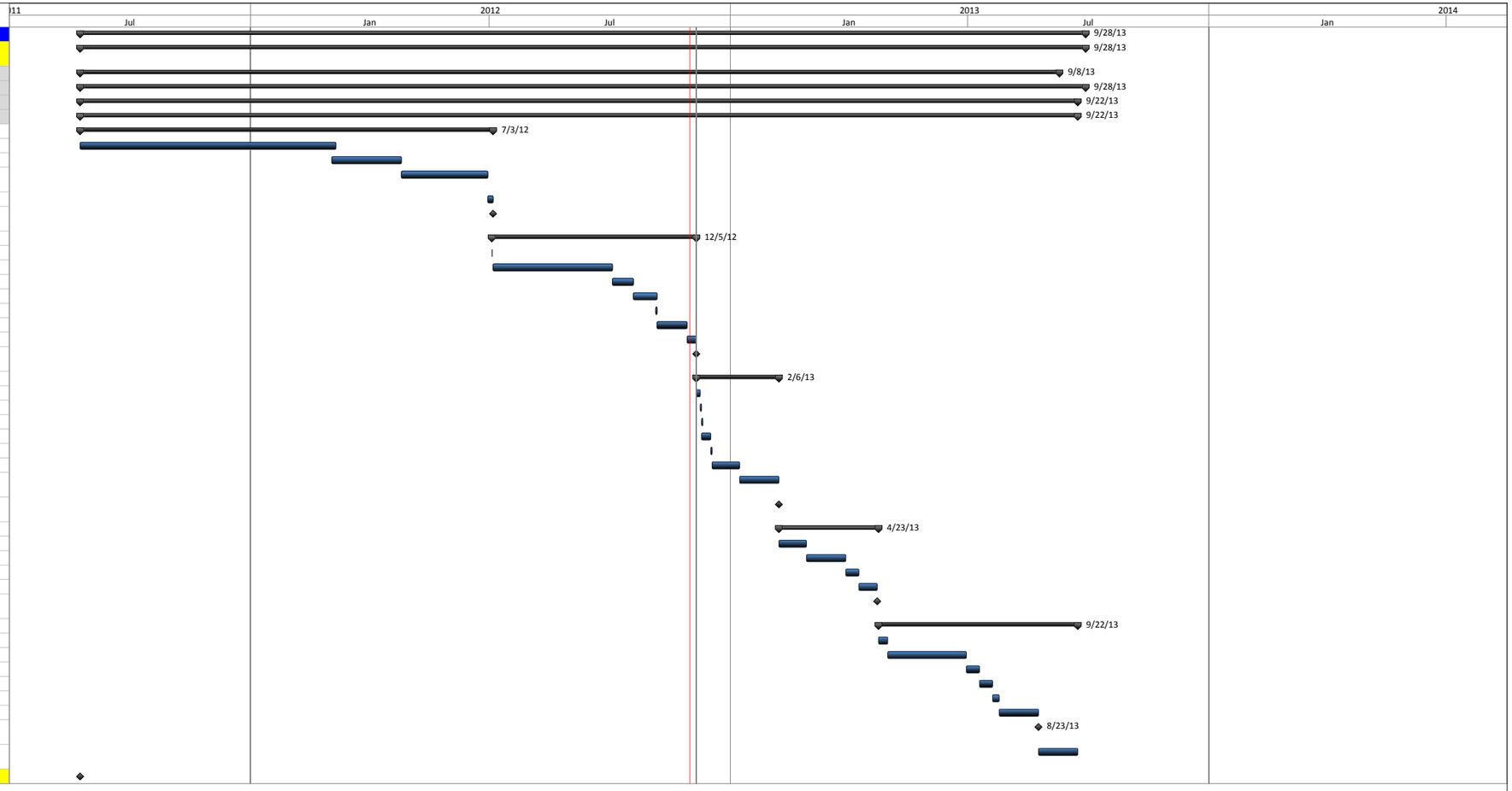
CS-C503 Location



Appendix B

Site-Specific Schedule

ID	CLIN	Task Name	Duration	Start	Finish
637	***0009P***	***SWMU 56 (SA-C056), OW-3640, SWMU-66, CS-C503***	***767 days***	***Wed 8/24/11***	***Sat 9/28/13***
638	0009PA	Phase 1 RI within 2 years from NTP: SA-C056, OW-C3640, SWMU 56, CS-C503	767 days	Wed 8/24/11	Sat 9/28/13
639		SWMU 56, Former Civil Engineering Storage Yard	747 days	Wed 8/24/11	Sun 9/8/13
679		OW-C3640 - Oil Water Separator 3640	767 days	Wed 8/24/11	Sat 9/28/13
719		SWMU 66 D-5 Hard fill Area Near AOC-27	761 days	Wed 8/24/11	Sun 9/22/13
759		CS-C503 PCB Contaminated Sediment in Retention Basin	761 days	Wed 8/24/11	Sun 9/22/13
760	0009PA.21	Draft UFP-QAPP	315 days	Wed 8/24/11	Tue 7/3/12
761	0009PA.21	Prepare Working Copy of UFP-QAPP	195 days	Wed 8/24/11	Mon 3/5/12
762	0009PA.21	USACE & AF Review/Comment on Working Copy	53 days	Sat 3/3/12	Tue 4/24/12
763	0009PA.21	Prepare Draft UFP-QAPP, Address Comments, Prepare Submittal Letter	66 days	Wed 4/25/12	Fri 6/29/12
764	0009PA.21	USACE & AF Approval of Draft UFP-QAPP	4 days	Sat 6/30/12	Tue 7/3/12
765	0009PA.21	Payment Milestone - USACE/AF Approval on Draft UFP-QAPP	0 days	Tue 7/3/12	Tue 7/3/12
766	0009PA.22	Final UFP-QAPP	156 days	Tue 7/3/12	Wed 12/5/12
767	0009PA.22	Produce/Submit Draft to Regulators	1 day	Tue 7/3/12	Tue 7/3/12
768	0009PA.22	Regulatory Review/Comment on Draft	91 days	Wed 7/4/12	Tue 10/2/12
769	0009PA.22	Prepare Draft Final UFP-QAPP	16 days	Wed 10/3/12	Thu 10/18/12
770	0009PA.22	USACE & AF Review of Draft Final	18 days	Fri 10/19/12	Mon 11/5/12
771	0009PA.22	Produce/Submit Draft Final	1 day	Mon 11/5/12	Mon 11/5/12
772	0009PA.22	Regulatory Concurrence of Draft Final	24 days	Mon 11/5/12	Wed 11/28/12
773	0009PA.22	Submit Final UFP-QAPP	7 days	Thu 11/29/12	Wed 12/5/12
774	0009PA.22	Payment Milestone - USACE/AF Approval and Regulator Concurrence on UFP-QAPP	0 days	Wed 12/5/12	Wed 12/5/12
775	0009PA.23	RI Field Work	63 days	Thu 12/6/12	Wed 2/6/13
776	0009PA.23	Field work Scheduling	3 days	Thu 12/6/12	Sat 12/8/12
777	0009PA.23	Mobilization	1 day	Sun 12/9/12	Sun 12/9/12
778	0009PA.23	Installation Briefing	1 day	Mon 12/10/12	Mon 12/10/12
779	0009PA.23	Complete Field work	7 days	Mon 12/10/12	Sun 12/16/12
780	0009PA.23	Demobilization	1 day	Mon 12/17/12	Mon 12/17/12
781	0009PA.23	Laboratory Analysis	21 days	Tue 12/18/12	Mon 1/7/13
782	0009PA.23	Tech Memo/Data Management/Validation/ERPIMS	30 days	Tue 1/8/13	Wed 2/6/13
783	0009PA.23	Payment Milestone - USACE/AF Approval of Tech Memo	0 days	Wed 2/6/13	Wed 2/6/13
784	0009PA.24	Draft Phase I RI Report	76 days	Thu 2/7/13	Tue 4/23/13
785	0009PA.24	Prepare Working Copy of Document	21 days	Thu 2/7/13	Wed 2/27/13
786	0009PA.24	USACE/AF Review/Comment on Working Copy	30 days	Thu 2/28/13	Fri 3/29/13
787	0009PA.24	Prepare Draft Document	10 days	Sat 3/30/13	Mon 4/8/13
788	0009PA.24	USACE/AF Approval of Draft Document	14 days	Tue 4/9/13	Mon 4/22/13
789	0009PA.24	Payment Milestone - USACE/AF Approval of Draft Report	1 day	Tue 4/23/13	Tue 4/23/13
790	0009PA.25	Final Phase I RI Report	152 days	Wed 4/24/13	Sun 9/22/13
791	0009PA.25	Produce/Submit Draft Document to Regulator	7 days	Wed 4/24/13	Tue 4/30/13
792	0009PA.25	Regulatory Review/Comment on Draft Document	60 days	Wed 5/1/13	Sat 6/29/13
793	0009PA.25	Prepare Draft Final Document	10 days	Sun 6/30/13	Tue 7/9/13
794	0009PA.25	USACE/AF Review of Draft Final	10 days	Wed 7/10/13	Fri 7/19/13
795	0009PA.25	Produce/Submit Draft Final	5 days	Sat 7/20/13	Wed 7/24/13
796	0009PA.25	Regulatory Concurrence of Draft Final	30 days	Thu 7/25/13	Fri 8/23/13
797	0009PA.25	Payment Milestone - USACE/AF Approval and Regulator Concurrence on Final Report	0 days	Fri 8/23/13	Fri 8/23/13
798	0009PA.25	Submit NFRAP supporting Documents or Tech Memo to establish DQOs	30 days	Sat 8/24/13	Sun 9/22/13
799	0009PB	Phase 1 RI within 2 years from NTP: SWMU 1 (not funded)	0 days	Wed 8/24/11	Wed 8/24/11



Progress | Task | Split | Milestone | Summary | Project Summary

Appendix C

Sampling Standard Operating Procedures

- SOP-1: Soil Sampling, #016-1510232, 02/10/2012
- SOP-2: Field Quality Control Samples, #011-1578758, 10/05/2012
- SOP-3: Field Documentation, #010-1578753, 10/05/2012
- SOP-4: Field Equipment Decontamination, #002-1578775, 10/05/2012
- SOP-5: Packaging and Shipping of Environmental Samples, #006-1510206, 02/10/2012
- SOP-6: Air Monitoring Instrumentation Manual, #007-52523V4, 04/11/2007
- SOP-7: Sample Custody, #004-1510208, 02/10/2012
- SOP-8: Classification and Description of Soil, Sediment, and Rock, #007-1577802, 10/02/2012
- SOP-9: Sediment Sampling, #019-1543704, 06/14/2012
- SOP-10: Investigation Derived Waste, #018-129394, 10/02/2012
- SOP-11: Groundwater Sampling, #009-1510481, 02/10/2012

Bay West

Standard Operating Procedure

Soil Sampling

CORP-ENV-016-1510232

Revised: February 10, 2012

Review and Approval:

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Date: 02/20/2012

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

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NOTE: This SOP is current as of the date printed on the bottom. Bay West personnel may produce paper copies of this procedure printed from the controlled-document electronic file located on the Intranet. However, it is their responsibility to ensure that they are trained and utilize the current version of this procedure.

1.0 INTRODUCTION

1.1 Purpose

The objective of this Standard Operating Procedure (SOP) is to define the techniques and requirements for collecting soil samples from both the surface soil and the unconsolidated zone. Techniques include the use of hand trowels, hand augers, Shelby tubes, split-spoon, and macro-core samplers.

Analytical procedures are often targeted as the main source of error in data analysis, but generally only represent a minimal contribution to the total error. Field errors are often the major source of error. Potential sources of field error are: sample collection, sample handling, transport, preparation, preservation, and sample identification. Some of these restrictions and/or limitations are discussed in detail in Section 7.1. The sampling portion of any data collection effort has historically been the most difficult to assess when assessing project data quality. Through the correct and consistent application of this SOP, Bay West field staff will generate samples which are both representative of the targeted sample location, and comparable to another data set assuming similar samples and sample conditions.

1.2 Scope

The scope of this SOP covers all aspects of soil sampling including but not limited to: surface soils, subsurface soils accessible from an excavation, stockpiles and samples collected from subsurface exploration devices such as a drill rig. Sediment sampling is not part of this SOP.

2.0 DEFINITIONS

Surface Soils or Surficial Zone – The layer of soil that comprises the top 10 – 12 inches of soil. This soil may be *disturbed soil*, that may be found in an industrial area that receives a great deal of traffic or *undisturbed soil*, such as forest soils that are fairly compact.

Unconsolidated Zone - The layer of soil below the surficial zone but above bedrock that exists in a relatively loose state.

Hand Trowel – a small, stainless steel device that is operated with one hand to scoop surface soil into a sampling container. A small shovel may also be used to collect surface soil samples.

Hand Auger - A stainless steel cylinder (bucket or tube) approximately 3 to 4-inches in diameter and one foot in length, open at both ends with the bottom edge designed to twist into the soil and cut out a soil core. The bucket or tube collects the soil sample. The auger has a T-shaped handle (for hand operation) attached to the top of the bucket by extendable stainless steel rod(s). A slide hammer can be attached, in place of the T-handle to drive a tube sampler.

Shelby Tube - A cylindrical sampling device generally made of steel, which is driven into the subsurface soil through the hollow-stem auger or hand auger device with a slide

hammer. The tube, once retrieved, may be capped and the undisturbed soil sample extruded in the laboratory prior to analysis.

Split-Spoon - A cylindrical sampling device generally made of carbon steel, which fits into a hollow stem auger. The split spoon is hinged lengthwise, which allows the sample to be retrieved by opening ("splitting") the spoon.

Macro-core – A piston rod sampling device, typically 4 or 5-feet in length, generally made of carbon steel, which fits onto hollow push rods. A push probe rig pushes the sampler to the desired sample depth then extension rods are lowered through the hollow push rods to release a stop-pin which allows the sampler to be filled when the sampler is advanced at the desired sampling interval.

Subsurface Soil - The soil which exists deeper than approximately one foot (30-cm) from the surface, but above bedrock or any other consolidated material.

Grab Sample - A discrete portion or aliquot taken from a specific location at a given point in time. Grab samples are not composited.

Liner - A cylindrical sampling device generally made of plastic, brass, stainless steel, or Teflon, that is placed inside a split-spoon, macro-core or hand auger bucket to collect soil samples.

Sample Syringe – Cut or open-end syringe used to core a measured quantity of soil that can be extruded into a sample jar that contains sodium bisulfate or methanol preservative.

EnCore® Sampler – A coring device that allows a specific quantity of soil to be collected (e.g. 5-grams and 25-grams). This device has a tight fitting cap that seals with an o-ring. Samples collected in this manner may be frozen prior to shipment to the lab.

Composite Sample - Two or more grab sub-samples taken from a specific media and site at a specific point in time. The sub-samples are collected and mixed, and then a single average sample is taken from the mixture.

Auger Flight - A steel section (typically 5-feet in length) attached to an auger to extend the auger as coring depth increases.

3.0 RESPONSIBILITIES

Sampler - The Sampler is responsible for the sampling, collection, labeling, analysis and recording of data as specified in this SOP.

Site Supervisor - The Site Supervisor is responsible for making sure that field personnel are trained in the use of the sampling procedure and the required equipment, and for making sure that soil samples are collected in accordance with the approved procedure and any other SOPs pertaining to specific media sampling.

Project Manager - The Project Manager is responsible for maintaining logbooks and forms and for approving techniques not specifically described in this SOP and documenting these techniques.

4.0 REQUIRED EQUIPMENT

This section provides a list of equipment that is generally used to collect soil samples but does not necessarily include all possible types of soil sampling equipment. Bay West SOPs, including specified equipment and associated forms, shall be obtained and followed for classification and description of unconsolidated material, decontamination, sampling, packaging and shipping. Specific SOPs are listed below:

- Classification and Description of Sediment and Rock. (SOP# CORP-ENV-007-65715, current version) Provides procedures for the classification and description of unconsolidated material (sediment) and rock samples collected during field operations at sites where environmental investigations are performed;
- Field Equipment Decontamination at Non-Radioactive Sites. (SOP# CORP-ENV-002-65422, current version) Presents procedures for decontamination of field sampling equipment. All equipment will be decontaminated between sampling locations to prevent cross contamination of samples;
- Packaging and Shipping of Environmental Samples. (SOP# CORP-ENV-006-1510206, current version) Establishes packaging and shipping requirements and guidelines for environmental sample shipping; and
- Sample Custody. (SOP# CORP-ENV-004-1510208, current version) Presents sample custody procedures to maintain and document sample possession that are traceable from the time the samples are collected until their analysis is complete. The data generated may be introduced as evidence in legal proceedings.

The following subsections identify the types of equipment used for a specific sampling method. Prior to initiating field work it will be necessary to determine the type of equipment that will be needed to complete the sampling. The selection and restrictions/limitations of equipment and sample methods are discussed in greater detail in Section 7.1. In addition to the SOPs identified above and the equipment specified in the following sections, the sampler will also obtain the following materials before initiating field work:

- Site-specific plans
- Field log book
- Indelible black ink pens and markers
- Labels and appropriate forms/documentation for sample shipment
- Clear, waterproof tape
- Laboratory prepared sample containers
- Preservatives
- Insulated coolers(s) and waterproof sealing tape
- Ice bags or "blue ice"
- Latex, Nitrile, or other appropriate soil sampling gloves as specified in the SSHP
- Plastic bags with a zipper closure

- Packing material such as "bubble wrap" for packing the sample containers in the coolers
- Stainless steel and/or Teflon-lined spatulas and pans, trays, or bowls
- Plastic sheeting
- Personal protective clothing (PPE) and equipment as specified in Site Safety Plan

4.1 Manual (Hand) Sampling

- Hand trowel or a small shovel
- Hand auger: flighted, bucket, or tube-type auger as required by the site-specific plans
- Extension rods, as needed
- Wrench(es), pliers, tool kit

4.2 Split-Spoon and Shelby Tube Sampling

- Drill rig equipped with a 140-lb drop hammer and sufficient hollow-stem augers to drill to the depths required by the site-specific plans.
- Sufficient numbers of split-spoon or Shelby tube samplers so that at least one is always decontaminated and available for sampling. Three split-spoon or Shelby tube samplers are generally the minimum necessary. (Shelby tubes are usually used only once.).
- Split-spoon liners, as appropriate.
- Wrench(es), hammer, tool kit
- Basket or spring retainers may be needed for split-spoon sampling in loose, sandy soils.

Note: Shelby tubes may not retain the sample in loose, sandy soils.

4.3 Macro-core Sampling

- Push-probe rig with sufficient push rods to probe to the depths required by site-specific plans
- Sufficient number of macro-core liners for the planned number of sampling intervals
- Hooked blade knife to cut the liners

5.0 PROCEDURES

5.1 Preparation

The following general steps will be followed in preparing to collect soil samples:

1. Don the appropriate PPE as dictated by the site-specific health and safety plan.
2. The collection points shall be specified in the site-specific plan, located on a site map, and referenced in the field logbook.

3. Processes for verifying depth of sampling must be specified in the site-specific plans.
4. Clear away vegetation and debris from the surface at the sampling location.
5. Prepare an area next to the sample collection location for laying out cuttings by placing plastic sheeting on the ground or over a work area, if necessary.
6. Set up a decontamination line, if decontamination is required.

5.2 Collection

The following general steps will be followed when collecting all surface or subsurface soil samples:

1. VOC samples or samples degraded by aeration shall be collected first and with the least disturbance possible. These samples shall be collected as grab samples using an EnCore® sampler, a Terra Core® sampler or a "cut syringe" sampling device. Immediately after sample collection, the EnCore® sampler will be sealed and placed into the foil bag it came with. If a Terra Core® sampler or a "cut syringe" sampling device is used, the soil will immediately be extruded into a 40ml VOA vial containing the appropriate preservative. The vial will be sealed and the level of the liquid preservative will be noted and it should be sufficient to completely cover the soil aliquot. If not, another sample should be collected. (Reference EPA Method 5035 from EPA SW-846, Test Methods for Evaluating Solid Waste).
2. Sampling information shall be recorded in the field logbook and on any associated forms. Describe the soil sample, according to Bay West Classification and Description of Sediment and Rock SOP (SOP# CORP-ENV-007-65715, current version), in the field logbook or on the Soil Boring Log Form.
3. Specific sampling devices to be used shall be identified in the site-specific field sampling plans.
4. Care must be taken to prevent cross-contamination and misidentification of samples.
5. Processes for verifying depth of sampling must be specified in the site-specific plans.
6. When all sampling is complete, dispose of cuttings, plastic sheeting, etc., as specified in the site-specific plans.
7. Complete the field logbook entry and other appropriate forms, being sure to record all relevant information before moving to the next location and/or leaving the site.
8. Decontaminate all equipment according to Bay West Field Equipment Decontamination at Non-Radioactive Sites SOP (SOP# CORP-ENV-002-65422, current version) before proceeding to the next sample location.
9. Properly package all samples for shipment and complete all necessary sample shipment documentation. Transfer custody of samples to the appropriate personnel. See Bay West Packaging and Shipping of Environmental Samples and Sample Custody SOPs (SOP# CORP-ENV-006-1510206, current version, and SOP# CORP-ENV-004-1510208, current version, respectively).

5.3 Manual (Hand) Augering

The following steps will be followed when collecting hand-augured samples:

1. Auger to the depth required for sampling. Place cuttings on plastic sheeting or as specified in the site-specific plans. If possible, lay out the cuttings in stratigraphic order.
2. Throughout the augering, make detailed notes concerning the geologic features of the soil or sediments in the field logbook.
3. Cease augering when the top of the specified sampling depth has been reached. If required, remove auger from the hole and decontaminate, or use a fresh auger. Then obtain the sample.
4. Collect a grab sample for VOC analyses (or samples that may be degraded by aeration) immediately and place in a pre-preserved sample container using a cut syringe or filling and sealing an EnCore® sampler.
5. Label the sample container with the appropriate information. Secure the label, covering it with a piece of clear tape.
6. Remaining sample will be homogenized for other analyses. The sample will be thoroughly mixed in a stainless steel bowl and small rocks and twigs will be removed prior to placing the sample in the appropriate container(s). Label container(s) as required.
7. Wipe exterior of containers clean with a Kimwipe® or paper towel.
8. Place containers in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler (4°C ± 2°C).
9. Proceed with further sampling, as required by the site-specific plans.

5.4 Manual (Hand) Augering Using a Tube sampler With Liner

The following steps will be followed when collecting hand-augured samples using a tube sampler with liner:

1. Auger to the depth required for sampling. Place cuttings on the plastic sheeting as specified in the site-specific plans. If possible, lay out the cuttings in stratigraphic order.
2. Throughout augering, make detailed notes concerning the geologic features of the soil or sediments in the field logbook.
3. Cease augering when the top of the specified sampling depth has been reached. Remove the auger from the hole and decontaminate.
4. Prepare a decontaminated tube sampler by installing a clean liner in the auger tube.

5. Use the slide hammer to advance and retrieve the sampler. Remove the liner from the tube and immediately cover ends with Teflon tape and cap the ends of the tube. Seal the caps with waterproof tape.
6. Label the sealed liners as required in the site-specific plans. Mark the top and bottom of the sample on the outside of the liner. Indicate boring/well number and depth on outside of liner.
7. Wipe outside of sealed liners clean with a Kimwipe® or paper towel.
8. Place sealed liners in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler (4°C ± 2°C).
9. Proceed with further sampling, as required by the site-specific plans.

5.5 Split-Spoon Sampling

The following steps will be followed when collecting split-spoon samples:

1. Remove any pavement and sub-base material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling, per Bay West SOP or the site-specific decontamination procedures.
3. Attach to the drill rig the hollow-stem auger with the cutting head, plug and center rod(s).
4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Remove the plug and center rods.
6. Install a decontaminated split-spoon on the center rod(s) and insert it into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the top of undisturbed soil or sediment.
7. Mark the center rod in 6-inch increments for 24-inches from the top of the auger flight(s).
8. Drive the spoon using the hammer. Use a full 30-inch drop for the hammer as specified by the American Society for Testing and Materials (ASTM) Method D-1586. Record the number of blows required to drive the spoon through each 6-inch increment.
9. Cease driving when the full-length of the spoon has been driven or upon refusal. Refusal occurs when little (<1-inch) or no progress is made for 50 blows of the hammer.
10. Pull the spoon free by using upswings of the hammer to loosen the sampler. Pull out the center rod and spoon.
11. Unscrew the split-spoon assembly from the center rod and place it on the plastic sheeting.

12. Remove the drive shoe and head assembly. If necessary, tap the split-spoon assembly with a hammer to loosen threaded couplings.
13. With the drive shoe and head assembly off, open (split) the spoon, being careful not to disturb the sample.
14. Label sample containers with appropriate information. Secure the label, covering it with a piece of clear tape.
15. If VOC analyses or analyses that may be degraded by aeration are to be conducted on the soil sample, collect a grab sample for the analyses immediately and place in a pre-preserved sample container using a cut syringe or filling and sealing an EnCore® sampler.
16. Record the sample identification number, depth from which the sample was taken, and the analyses to be performed on the samples in the field logbook and on the appropriate forms.
17. Remaining soil samples should be homogenized prior to placing samples in appropriate containers. Label containers as required.
18. Wipe outside of sample containers clean with a Kimwipe® or paper towel.
19. Place sample containers in zip-top plastic bags and seal the bags. Pack samples in a chilled cooler (4°C ± 2°C).
20. Continue to advance the hollow stem augers to the next sampling point. Collect samples as outlined above.
21. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.

5.6 Split-Spoon Sampling Using Liners

The following steps will be followed when collecting samples with lined split spoons:

1. Remove any pavement and subbase material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling.
3. Attach the hollow-stem auger with the cutting head and center rod(s).
4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Remove the plug and center rods.
6. Install a clean liner in the split-spoon barrel.
7. Install a decontaminated split-spoon on the center rod(s) and insert into the hollow-stem auger. Connect the hammer assembly and lightly tap the rods to seat the drive shoe at the top of undisturbed soil or sediment.
8. Mark the center rod in 6-inch increments for 24-inches from the top of the auger flight(s).
9. Drive the spoon using the hammer. Use a full 30-inch drop for the hammer as specified by ASTM Method D-1586. Record the number of blows required to drive the spoon through each 6-inch increment.
10. Cease driving when the full-length of the spoon has been driven or upon refusal. Refusal occurs when little (<1-inch) or no progress is made after 50 blows of the hammer.
11. Pull the spoon or tube free by using upswings of the hammer to loosen the sampler. Pull out the center rod and spoon.
12. Unscrew the split-spoon assembly from the center rod and place it on the plastic sheeting.
13. Remove the drive shoe and head assembly. If necessary, tap the split-spoon assembly with a hammer to loosen threaded couplings.
14. With the drive shoe and head assembly off, open (split) the spoon and remove the liner without disturbing the sample.
15. Wipe the outside of the sealed liners clean with a Kimwipe® or paper towel.
16. Mark the top of the core and the sample depth using a marker on the outside of the plastic liner.
17. Collect a grab sample for VOC analyses (or samples that may be degraded by aeration), if required, immediately and place in a pre-preserved sample container using a cut syringe or filling and sealing an EnCore® sampler. Pack samples in a chilled cooler. Collect headspace and other samples as required by the site-specific plans.

18. In the field logbook and on the boring log, describe sample lithology by observing cuttings and the bottom end of the sample in the liner.
19. Continue to advance the borehole to the next sampling point. Collect samples as outlined above.
20. When sampling is complete, remove the drilling rig to the heavy equipment decontamination site.

5.7 Macro-core sampling

The following steps will be followed when collecting samples with lined split spoons:

1. Plastic liner and solid drive point is placed in macro-core.
2. Macro-core is placed on the end of the push rods and driven to the desired sampling depth.
3. Extension rods are lowered through the hollow push rods to release a stop-pin in the drive point which allows the sampler to be filled when the sampler is advanced.
4. The macro-core is advanced through the desired sample interval.
5. The macro-core is retrieved by pulling the push rods.
6. The macro-core is opened and the plastic liner, filled with the soil sample, is removed.
7. Wipe the outside of the sealed liners clean with a Kimwipe® or paper towel.
8. Mark the top of the core and the sample depth using a marker on the outside of the plastic liner.
9. Immediately collect VOC samples by cutting the plastic liner and using a coring device as previously described to collect the sample. Pack samples in a chilled cooler. Collect headspace and other samples as required by the site-specific plans.
10. In the field logbook and on the boring log, describe sample by observing cuttings and the bottom end of the sample in the liner.
11. Continue to advance the cleaned and relined macro-core to the next sampling point. Collect samples as outlined above.
12. When sampling is complete, push rods need to be decontaminated before reuse or leaving the site.

5.8 Shelby Tube Sampling

The following steps will be followed when collecting samples using the Shelby tube:

1. Remove any pavement and sub-base material from an area of twice the bit diameter, if necessary.
2. The drilling rig will be decontaminated at a separate location prior to drilling.
3. Attach the hollow-stem auger with the cutting head, plug, and center rod(s).
4. Begin drilling and proceed to the first designated sample depth, adding auger flight(s) as necessary.
5. Slightly raise the auger flight(s) to disengage the cutting head, and rotate the auger without advancement to clean cuttings from the bottom of the hole.
6. Remove the plug and center rods.
7. Attach a head assembly to a decontaminated Shelby tube. Attach the Shelby tube assembly to the center rods.
8. Lower the Shelby tube and center rods into the hollow-seam augers and seat it at the bottom. Be sure to leave 30 inches or more of center rod above the lowest point to the hydraulic piston's extension.
9. Use the rig's hydraulic drive to push the Shelby tube into undisturbed soil. The tube should be pushed with a steady force.
10. When the Shelby tube has been advanced it's full-length or to refusal, back off the hydraulic pistons. Attach a hoisting plug to the upper end of the center rod, twist to break off the sample, and pull the apparatus out of the hole with the rig winch.
11. Retrieve the Shelby tube to the surface, detach it from the center rod, and remove the head assembly.
12. Since the typical intent of Shelby tube sampling is for engineering purposes and undisturbed sample is required, the tube ends should be sealed immediately, the top and bottom ends of the tube marked, and the tube should be transported to the laboratory in an upright position. Indicate boring/well number and depth on outside of liner.
13. Wipe the outside of the sealed tubes clean with a Kimwipe® or paper towel.
14. Place sealed tubes in zip-top plastic bags and seal bags. Pack samples in a chilled cooler (4°C ± 2°C).
15. Continue to advance the hollow stem augers to the next sampling point. Collect samples as outlined above.
16. When sampling is complete, remove the drilling rig to the heavy equipment decontamination area.

6.0 DOCUMENTATION

Individuals performing the work will record their observations in a commercially available, bound field logbook and the Boring Log Forms. Editing of field logbook data is not allowed. The source of the reference material or field charts used must be recorded in the field logbook. If a log book is used to record the sample description, the readings must be transferred within 24 hours to a Boring Log Form. The following is a general summary of the information to be documented:

- List of all personnel present
- Field conditions including:
  - Air temperature;
  - Wind speed/direction
  - Precipitation/moisture at the time of sampling event;
  - Ambient odors: and
  - Airborne dust.
  - Description of any exceptions to this SOP; and for each sample location
  - Sample Name used to identify the sample;
  - Sample method and equipment used;
  - Date and time of sample collection;
  - A list of all samples sent to each laboratory; and
  - Complete a Boring Log Form for all soil borings.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

7.1 Restrictions/Limitations

Only qualified persons trained in performing the requirements and the duties described in the SOP shall conduct the work. The Project Manager or QA/QC Manager will have the authority to decide whether or not an individual is qualified.

7.1.1 Selection and Limitations of Sampling Equipment

Choosing the proper sampling tool depends on the nature of the soil to be sampled and the goals of the project. Scoops, trowels and triers are used for surface grab samples (0 to 0.5 feet below grade) whereas the hand auger can be used to depths as great as 12 feet. The auger will produce a composite sample of the penetrated section.

The Shelby tube will collect a sample with the least amount of disturbance; however, this method precludes logging of the sample and is difficult to use in very well-compacted sediments. Using a continuous split-spoon sampling device or push probe macro-core is a rapid method of obtaining relatively undisturbed samples; however, depth of sampling is dependent upon sediment cohesiveness. Insufficient cohesiveness will result in poor sample recovery and borehole collapse. A split-spoon or macro-core are good general purpose sampling devices.

7.1.2 Removal of Extraneous Material

If indicated in the project planning documents, extraneous material is removed at the time of sample collection. If rocks, debris, root, twigs, etc. are not removed in the field, there may be greater chance for inconsistent sub-sampling in the laboratory and/or submitting insufficient sample volume. In some cases this material may have to be saved and analyzed separately depending on the project requirements.

Obtaining an aliquot from a sample container is referred to as laboratory subsampling and is performed by laboratory personnel during sample preparation steps. Current SW-846 and other standard reference methods provide little or no guidance on subsampling within their preparatory methods and laboratory staff commonly overlooks these critical procedures. Furthermore, it is common for many analytical methods to require only a fraction (1 to 30-grams for soils) of the submitted sample to be subjected to the actual analysis. Because only a fraction of the submitted sample is actually involved in the evaluation of the sampling location, it is important that the entire sample be truly representative. Adequate sample homogenation prior to laboratory submittal will increase the degree of sample representativeness.

7.1.3 Sample Homogenizing

Proper homogenizing technique is critical to accurately assessing the condition of a site, and to generating QC data that reflects the quality of the environmental data produced. Depending on site characteristics and the extent of contamination, the project plans may address homogenizing techniques directly. The generation of field control samples (e.g., replicate samples) will provide a mechanism for evaluating matrix heterogeneity and the sampling and handling techniques of the field personnel. As always homogenization techniques should not be used for VOC analysis or other parameters that require undisturbed sample collection.

Obtaining samples in a soil or sediment matrix requires homogenization of the sample aliquot prior to filling sample containers (with exception of VOC type parameters). Before sample mixing is performed, instructions on the removal of extraneous sample materials (grass or materials in "root zone," leaves, sticks, rocks, etc.) should be reviewed. Moisture content, sediments, and waste materials may inhibit the ability to achieve complete mixing prior to filling sample containers. However, it is extremely important that solid samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample location.

Homogenization procedures may be accomplished by several methods. The method best suited for the media will depend on the physical characteristics of the solid material (e.g., heterogeneity of media, maximum particle size present, moisture content, etc.). In general, homogenization is accomplished by filling a properly decontaminated container with the sample and mixing it with a decontaminated implement. The container should be large enough to hold the sample volume and accommodate the procedures without spilling. In most cases, the method of choice for mixing is referred to as cone and quartering and can be performed in a bowl or tray of an appropriate material (depending on the analytical parameters to be performed). If the solid medium is not amenable to

cone and quartering techniques due to the high moisture content or high cohesiveness of the waste, alternative procedures may need to be pursued, i.e., kneading, particle size reduction, or particle size separation devices (i.e., sieves). Additional procedures will likely require expansion of the field decontamination scope.

7.1.4 Compositing Samples

There are various types of composite sampling techniques available. The technique of compositing discrete samples is typically employed when the site under investigation is quite large to improve the precision (lower the variance) of the estimated average contaminant concentrations, especially when contamination exhibits a short-range heterogeneity, and to decrease the probability of making a wrong decision based on limited data. Discussion between project planners and data users should be done to determine the appropriateness of applying compositing schemes to meet project objectives. Composite sampling is not specific to one matrix. Rather it can be utilized for solid, semisolid, liquid, and air matrices.

Composite samples consist of a series of discrete grab samples that are mixed together to characterize the average composition of a given material. The discrete samples used to make up a composite sample are typically of equal volume. Discrete samples must be collected in an identical fashion. Likewise, the number of grab samples forming a composite should remain consistent (i.e., a number and pattern for collection of grab samples within a grid should be selected and, for a given grid size, should not be changed). Homogenizing techniques must be used on each individual discrete sample, and the resultant composite sample.

If a contaminant is detected in a composite sample, each discrete grab sample that made up the composite should be analyzed individually to determine actual distribution of the contamination. Therefore the discrete samples should be initially submitted to the laboratory with the composites on "hold" for storage and potential analysis.

Potential analytical problems associated with compositing is when the sample matrix is not amenable to mixing techniques, which resulting in a non-homogeneous sample mixture compromising the representativeness of the composite results.

Areal Composite - Areal composite samples are samples collected from individual grab samples collected in an area or on a cross-sectional basis. Areal composites are made up of equal volumes of grab samples where all grabs are collected in an identical manner. Areal composite sampling is typically used for estimating average contaminant concentrations in surface soils or sediments. This is especially useful when contaminants are present in a nugget form (i.e., TNT chunks, lead shot, etc.), exhibiting large differences in concentration in a small area (short-range heterogeneity). Grid sizes should be kept moderate (<5 to 10-feet) in diameter, if project objectives and intended use of the data are to maintain aspects of a "discrete" sample while providing better overall coverage.

Vertical Composite - Vertical composite samples are also collected from individual grab samples but taken from a vertical cross section. Vertical composites are also made up of equal volumes of grab samples where all grab samples are collected in an identical

manner. Vertical profiles of soil borehole or sediment columns are examples of vertical compositing.

7.1.5 VOC Sample Preservation

Preservation of VOC samples in the field is critical to successful soil sampling. Generally, there are four methods to accomplish proper preservation: Using a sealing device, e.g. an EnCore® sampler or using one of three liquid preservation methods as provided by the laboratory. All four techniques can be successfully used, but they all have their drawbacks. The EnCore® sampler is probably the easiest technique to use in the field. EnCore® samples come in two sizes: 5 gram and 25 gram. The site specific SAP will specify which one to use. Two of the specific EnCore® samplers should be collected for each sampling location. The sampler is pressed into the soil until the small o-ring is observed in the tool window, the open end of the sampler is cleaned of any soil adhering to the sealing surface and the cap is then locked into place. The drawbacks are these samplers are expensive and are one time use only. The lab needs to extrude the sample upon receipt (with 48 hours) and place the soil into a preservative, an extra step on their part and a (small) potential step to spill or contaminate the sample.

The three field liquid preservation methods consist of the lab providing either: three 40ml *low level* VOA vials for each sample to be collected (Sodium Bisulfate and methanol preservatives), three 40ml *low level* VOA vials for each sample to be collected (Deionized water and methanol preservatives), or two 40ml *medium level* VOA vials for each sample to be collected (methanol preservative only). The preservation method depends on the concentration range of the analytes to be determined and will be specified in the site specific SAP.

The first field liquid preservation method (low level) requires two vials containing ~5ml of sodium bisulfate ($NaHSO_4$) solution and a small stir bar and one vial containing ~5ml of lab grade methanol. All of the vials are pre-weighed by the lab before shipment. The sampler uses a coring device, such as a Terra Core® or a cut syringe, to place approximately 5 grams of soil into each vial. Be certain the threads of the vial are free of soil before sealing the vial. It is not critical to know the exact mass of the soil added to the vial as the lab will weigh the vial prior to analysis. It is critical the liquid in the vial covers the top of the soil aliquot for proper preservation.

The second field liquid preservation method (low level) requires two vials containing ~5ml of Deionized (DI) water and one vial containing ~5ml lab grade methanol. All of the vials are pre-weighed by the lab before shipment. The sampler uses a coring device, such as a Terra Core® or a cut syringe, to place approximately 5 grams of soil into each vial. Be certain the threads of the vial are free of soil before sealing the vial. It is not critical to know the exact mass of the soil added to the vial as the lab will weigh the vial prior to analysis. It is critical the liquid in the vial covers the top of the soil aliquot for proper preservation.

The third field liquid preservation method requires two vials containing ~10ml of lab grade methanol. All of the vials are pre-weighed by the lab before shipment. The sampler uses a coring device, such as a Terra Core® or a cut syringe, to place approximately 10 grams of soil into each vial. Be certain the threads of the vial are free of soil before sealing the

vial. It is not critical to know the exact mass of the soil added to the vial as the lab will weigh the vial prior to analysis. It is critical the liquid in the vial covers the top of the soil aliquot for proper preservation.

Drawbacks to the field liquid preservation techniques are: spillage of the preservative, which renders the vials useless as the tare weight is lost, adding too much soil to the vial so the preservative does not cover all of the soil in the vial, and limited shelf life of the pre-preserved vials. Laboratories usually prefer one of the field liquid preservation methods because, after weighing the vial, the sample can often be placed directly on the instrument auto sampler for analysis. In addition, if no other sample jars are required for this particular sampling location, a small 250ml sample jar without preservative will need to be sent to the lab in addition to the liquid preserved vials for a percent moisture determination. Some laboratories may send a different number of vials according to their lab SOP. These differences will be detailed in the site specific SAP.

When performed properly, all four of these techniques will produce a valid, preserved soil sample. These soil sampling techniques are described in detail in EPA Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, as referenced below.

8.0 REFERENCES

American Society for Testing and Materials, *Penetration Test and Split Barrel Sampling of Soils*, Standard Method D-1586-84, 1984.

U.S. Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements for Field Methods*, DOE/HWP-69/R1, July 1990.

U.S. Department of Energy, Hazardous Waste Remedial Actions Programs, *Standard Operating Procedures for Site Characterizations*, DOE/HWP-100, July 1990.

U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste,* SW-846 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples. Final Update IV, Revision 6, February 2007.

Bay West

Standard Operating Procedure

Field Quality Control Samples

CORP-ENV-011-1578758

Revised: October 5, 2012

Review and Approval:

Developed by: _____ Date: October 5, 2012
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QA/QC Manager
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Reviewed by: _____ Date: October 5, 2012
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Approved by: _____ Date: October 5, 2012
Signature
Vice President of Operations
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Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the Quality Assurance/ Quality Control (QA/QC) Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

This Standard Operating Procedure (SOP) describes the requirements for the collection of field Quality Control (QC) samples to ensure the reliability and validity of field and laboratory data. Field QC samples shall be collected as described in the SAP/QAPP and sent to the pre-approved subcontract laboratory for analysis.

1.2 Scope

This SOP shall be implemented by all Bay West Project Managers when collecting field QC samples to meet the quality control requirements for the project. **Note:** Subcontractors performing work under any Bay West Project shall follow the elements of this SOP for collecting field QC samples to ensure the reliability and validity of field and laboratory data for the Project. The subcontractor may use their own procedure(s) as long as the substitute meets the requirements prescribed by the Bay West Project SAP/QAPP, and is approved by the Bay West Project's Manager and the Quality Assurance/Quality Control Manager before sampling activities begin.

2.0 DEFINITIONS

Aliquot – An aliquot is a portion of a sample that is representative of the entire sample.

Ambient Blank – Ambient blanks are prepared only when specified by project-specific requirements and only when volatile organic compound (VOC) samples are taken for analysis. The ambient blank consists of reagent grade water poured into a VOC sample vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis.

Ambient blanks are used to assess the potential introduction of contaminants from ambient sources (e.g., gasoline motors in operation, etc.) to the samples during sample collection.

Background Sample – A sample collected from an area or site similar to the one being studied, but located in an area known or thought to be free from contaminants of concern.

Data Quality Objectives (DQOs) – DQOs are qualitative and quantitative statements that clarify the study objectives, define the most appropriate type of data to collect, determine the appropriate conditions from which to collect the data, and specify tolerable limits on decision errors that will be used as the basis for establishing the quantity and quality of data needed to support the decision.

It is imperative that the DQOs be defined in the site SAP(s) and/or QAPP(s) prior to the initiation of field and analytical laboratory work. The responsible parties performing the

work must be aware of the DQOs so that informed decisions during the course of the project can be made to attain those DQOs.

Equipment Blank (EB) – A sample of reagent grade water usually supplied by the laboratory (deionized water or DI water, NOT tap or store-purchased drinking water) poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. EBs shall be collected immediately after the equipment has been decontaminated and prior to sampling. EBs are used to assess the effectiveness of Bay West equipment decontamination procedures.

The frequency of collecting EBs shall be specified in project planning documents. If project specific guidance is not provided, collect a minimum of one EB per equipment setup per sampling day in the field. The blank is typically analyzed for all laboratory analyses requested for the environmental samples collected at the site.

Field Duplicate or Blind Field Duplicate Sample – Duplicate samples are two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are generally a single sample homogenized and divided into equal parts for analysis, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned a unique identification number in the field by Bay West field staff. When blind field duplicate samples are required, the sample identification is disguised to the laboratory by altering sample names and/or sample times. Indications of non-homogeneous samples should be clearly documented in the field notes. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Split samples, sent to different laboratories, are considered duplicate samples.

Field Replicates – A field replicate sample is two or more samples representing the same population characteristic, time, and place, which are independently carried through all steps of the sampling and measurement process in an identical manner. These samples are not homogenized but collected in rapid succession from the same location. Samples for VOC analysis are always replicate samples (though often misnamed “duplicates”). For example, VOC vials are filled in succession and not mixed; EnCore® soil samples are collected next to one another, the soil is not shared between containers. Specific locations are usually designated for collection of field replicate samples in the SAP/QAPP. Replicate samples are used to assess total (sampling and analysis) method variance and precision.

Matrix Spike (MS) and Matrix Spike Duplicates (MSD) – The MS and MSD are an aliquot of an actual sample spiked in the laboratory with known concentrations of all analytes listed in the method and at project-specific QC concentration limits. With the exception of VOC samples, MS/MSD samples are essentially duplicate samples until

they reach the laboratory. If not otherwise specified, each analyte in the MS and MSD shall be spiked at a level less than or equal to the midpoint of the analytical calibration curve for each analyte. The location of the MS and MSD samples will be specified in the SAP/QAPP and clearly designated on the Chain of Custody (CoC) by Bay West field personnel.

The MS and MSD are used to document the accuracy/bias of a method due to the sample matrix associated with the project. MS and MSDs will be analyzed at a rate of 5% (one spike per 20 samples and one spike duplicate per 20 samples) or as specified in the SAP/QAPP. The performance of the MS and MSD are evaluated against the method and project-specific QC acceptance limits and appropriate analytes (in all related samples) shall be qualified according to the data qualification criteria. The MS recoveries from samples that contain significant native background levels of the target analytes will be evaluated using the professional judgment of the Bay West Project Chemist for acceptance. Significant native background level is defined as an amount equal to or above that concentration which the laboratory spiked into the sample.

Matrix spike recoveries and relative percent differences will be calculated and reported for all laboratory spike pairs along with any analyte/matrix specific control limits.

Quality Control Samples – Samples used in a planned check of the operation of a measurement system to obtain a measure of the quality of the data generated.

Sampling and Analysis Plans– SAPs are documents describing the work to be performed on a site. SAPs are generally an attachment to work plans and delineate the sampling requirements of the task. SAPs may also consist of Quality Assurance Project Plans and Field Sampling plans, as required by the client.

Site-Specific Health and Safety Plan (SSHP) – A SSHP that is specific to a site or Emergency Response (ER)-related field activity that has been approved by the health and safety manager. This document contains information specific to the project including scope of work, relevant history, descriptions of hazards by activity associated with the project site(s), and techniques for exposure mitigation (e.g., personal protective equipment and hazard mitigation).

Temperature Blank – Consists of a vial filled in the laboratory with water, placed in sample coolers before or during sample collection to ensure temperature equilibration with the samples and returned to the laboratory. Upon receipt of samples, the laboratory will determine and record the temperature of the samples upon receipt using the temperature blank.

Trip Blank – Consists of two 40 ml VOC vials filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample, and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed

only for VOC analytes (In some cases low level PAH samples will require trip blanks. This shall be specified in the project-specific documents.)

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis of VOCs shall contain one trip blank set unless otherwise specified by the client. For methanol preserved soil samples being analyzed for gasoline range organics (GRO) or VOCs, a methanol blank shall be utilized as the trip blank. At least one trip blank set is required for all field events that include the collection of samples for volatile organic compound analysis.

3.0 RESPONSIBILITIES

Sampler/Field Team Member – The sampler or field team members implementing this procedure should have a general knowledge of the Environmental Protection Agency (EPA) methods and protocols for sampling. The sampler is responsible for the sampling, collection, labeling, analysis, and recording of data as specified in this SOP.

Site Supervisor – The Site Supervisor is responsible for coordination of all field activities. These include, but are not limited to, adhering to the site project plans, ordering the required field paperwork (sample collection logs and chains of custody), arranging for the correct analytical methods, obtaining the correct bottles, labels, and coolers, arranging the field team efforts, and providing the screening results for shipment/transport requirements.

The Site Supervisor is responsible for adherence to sampling protocols mandated by all applicable regulations and analytical methods as described in the site project plans. When ordering field paperwork, the Site Supervisor shall ensure that the field QC sample requirements are included.

The Site Supervisor is also responsible for ensuring that field team members who are involved in the collection of field QC samples are familiar with the objectives of and properly trained on each sampling procedure to be used.

Project Manager – The Project Manager is responsible for ensuring that the project plans are complete, reviewed by the appropriate personnel, and approved. The Project Manager is also responsible for approving techniques not specifically described in this SOP.

4.0 REQUIRED EQUIPMENT

Equipment required to implement this procedure shall be consistent with project requirements as listed in the site SAP/QAPP.

5.0 PROCEDURES

5.1 Pre-Operation Activities

The requirements for field QC samples must be evaluated as part of the preparation of site SAP/QAPP. Field QC sample requirements are provided in the following table. In addition to the field QC samples listed in the table, certain projects may require other types of samples be collected to obtain information (e.g., background and control samples). These types will be specified in the site SAP/QAPP.

Table 5-1 Field QC Sample Requirements

QC Sample Type	Sample Matrix	Frequency	Purpose
Equipment Rinsate Blank	Deionized water used to rinse equipment	One per day per equipment setup	To evaluate decontamination procedures
Field Duplicate or Blind Field Duplicate	All	One per day per matrix type or one per 10 samples, whichever is more frequent	To evaluate sampling and analysis reproducibility
MS and MSD	All	One set of MS and MSD per matrix type and per 20 samples.	To evaluate the effect of the sample matrix on accuracy and precision
Temperature Blank	All	One in each cooler	To evaluate sample conditions upon receipt at the laboratory
Trip Blank	VOC soil – Methanol VOC water – deionized water	One for each cooler containing VOC samples	To determine contamination during storage and transport

Obtain deionized water in sealed containers appropriate for transport to the field and in sufficient quantity to prepare the required equipment rinsate blanks. Do not use tap water or drinking water purchased from a local store, since these sources typically contain trihalomethanes.

Trip blanks are required for all field events that include the collection of samples for VOC analysis unless otherwise specified by the client. Trip blanks are provided by the laboratory and must be prepared at the beginning of the sampling activities and stored with the regular sample containers during the entire project. The containers should be preserved, filled, and sealed at the laboratory. Trip blanks for soil sampling activities shall be prepared using VOC-free methanol or as required by the SAP/QAPP.

5.2 QC Collection Procedures

QC samples shall be collected and prepared in the manner defined below. **Table 5-1** shows the collection frequency of field QC samples that shall be addressed in the site specific SAPs/QAPPs.

5.2.1 Equipment Blank

After decontaminating the field sampling equipment, rinse with deionized water and collect the rinsate for analysis. Assure that all equipment surfaces that come in contact with the sampling materials are rinsed (e.g., the inside of a bailer). The rinsate water should be collected immediately after decontamination and prior to sample collection. Note: Do not collect the water used for cleaning/decontaminating the field sampling equipment as your EB Sample.

5.2.2 Field Duplicate or Blind Field Duplicate

At the frequency specified in **Table 5-1**, collect two separate samples from the same source and at the same location and time. Place the samples in separate containers, follow the sample preservation procedure, label each as a unique sample, and submit both samples for the same analyses.

On rare occasion, samples used for field duplicates may have problems with homogeneity (e.g., suspended solids, non-aqueous phase liquid [NAPL], etc.). In these cases the samples should be homogenized as best as possible and the sample conditions noted in the field logbook. This should not be done for VOC samples due to possible loss of analytes during homogenizing.

Note: blind field duplicates require disguised sample identification to the laboratory.

5.2.3 Matrix Spike and Matrix Spike Duplicate

At the frequency specified in **Table 5-1**, collect three separate samples (parent, MS, and MSD) from the same source and at the same location and time. Place the samples in separate containers, follow the sample preservation procedure, label the first sample bottle (the parent field sample) according to standard project identification procedure in order to have the laboratory analyze the sample and report sample concentrations. The remaining two samples should contain the same field sample identification in addition to the text “MS” on one sample, and “MSD” on the second sample. The CoC can list the parent sample identification on one line and the following two lines should list the same parent sample identification and the text “MS” and “MSD” or the three samples can be listed on one line with the appropriate notation listed on the CoC. Each laboratory may have different procedures to reach the end goal. Either technique will provide the laboratory with the appropriate information. Additional detail on sample labeling can be found in **Section 6.0**.

On rare occasion, samples used for the parent sample, and MS and MSD samples may have problems with homogeneity (e.g., suspended solids, NAPL, etc.). In these cases all three samples should be homogenized as best as possible and the sample conditions noted in the field logbook. This should not be done for VOC samples due to possible loss of analytes during homogenizing.

In order to determine MS and MSD percent recovery data the laboratory must:

1. Analyze the parent sample to determine the background analyte concentrations which are already present in the sample. (This requires the typical number of bottle(s) for a sample);
2. Use the MS sample bottle(s) for an MS by spiking known analyte concentrations into the sample bottle prior to analysis and analyzing it along with the entire sample batch; and
3. Use the MSD sample bottle(s) for an MSD by spiking known analyte concentrations into the sample bottle prior to analysis and analyzing it along with the entire sample batch.

Once all of the above three units are analyzed, the lab will subtract the background levels in the parent sample (these were determined by analyzing the sample in step #1) from the MS and MSD results. This is the reason the laboratory must know which sample is the parent sample. Once the MS and MSD results are corrected for the original sample levels, they are divided by the amount spiked and multiplied by 100 to calculate percent recovery data.

Ordering glassware/bottles and collecting the correct number of sample bottles can be tricky depending on the analysis performed and the individual laboratory's procedure. For examples see **Table 5-2**.

Table 5-2 MS/MSD Bottle Requirements Example for Water Samples

Analysis	Number of bottles requested from Lab	Requirements for sample	Requirements for MS sample	Requirements for MSD sample
VOCs sample only	Three 40-ml vials total	3 vials	None	None
VOC sample with MS/MSD samples	Nine 40-ml vials total	3 vials	3 vials	3 vials
DRO sample only	Two 1-Liter bottles total	1 bottle minimum, 2 bottles preferred	None	None
DRO sample with MS/MSD samples	Six 1-Liter bottles total	1 bottle minimum, 2 bottles preferred	1 bottle minimum, 2 bottles preferred	1 bottle minimum, 2 bottles preferred
Metals sample	One 250-ml plastic	50 ml from bottle	None	None
Metals sample with MS/MSD	One 250-ml plastic (no extra bottles required)	50 ml from bottle	50 ml from parent bottle	50 ml from parent bottle

The number of bottles required to perform MS and an MSD can range from tripling the normal requirement to no extra bottles. When faced with difficult or onerous sampling conditions, bottle requirements can be reduced with laboratory and the Bay West Project Chemist's prior approval.

5.2.4 Temperature Blank

Specify on the bottle order that all coolers be shipped with a labeled temperature blank. If temperature blanks are not included in a bottle order they can be prepared by filling a 40-ml VOC vial with water and labeling appropriately. Upon receipt, the laboratory must record the temperature of the vial on the CoC.

5.2.5 Trip Blank

Trip blanks must be prepared by the laboratory before the day's sampling events and submitted with the regular samples at the end of each day's sampling activities (when collecting samples for VOC analysis), or at the end of the project if the required frequency is maintained. The number of trip blanks to be prepared depends upon the number and frequency of VOC samples to be collected (See **Table 5-1** for guidance). Maintain the trip blank containers with the regular sample containers throughout the sampling event and return them to the laboratory with the collected samples. Do not open the trip blanks.

6.0 DOCUMENTATION

All notes and comments associated with the QC samples will be recorded in a project specific field notebook in accordance with the Bay West SOP for Field QC Samples.

Field sampling personnel will properly identify all QC samples taken in the field with an adhesive sample label attached to each sample container. The sample label will contain the site name, field identification number, the date, time, and location of the sample collected, and identification of preservatives used. Sample information will be legibly printed with waterproof ink. The sample identification numbers will be recorded on field sheets, chain-of-custody forms, and other documentation records. The following are examples of field identification numbers for each type of QC sample:

- Equipment Blank: **Site Name-W-EB(date)**.

Blind duplicate samples will be numbered in the same sequence as the rest of the samples:

- Blind duplicate from a monitoring well sample: **Site Name-W-MW1**.

For blind duplicates, no indication that a sample is a duplicate will be provided on the sample label or the chain-of-custody form. The sample collection time will be altered on the chain-of-custody to mask identification as a duplicate. The location of blind duplicates will be listed in the field logbook.

- Matrix Spike: **Site Name-W-Sample Name (MS)**.
- Matrix Spike Duplicate: **Site Name-W-Sample Name (MSD)**.
- Trip Blank: **Site Name-W-TB(date)**.

The first letter character identifies the site name. The second letter character signifies the matrix sampled. The third set of number characters signifies the sample number and the type of QC sample.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

This SOP is to be used in conjunction with approved site SAPs/QAPPs. Only qualified persons trained in performing the requirements and the duties described in the SOP shall conduct the work. The Project Manager or QA/QC Manager will have the authority to decide whether or not an individual is qualified.

All proposed Bay West site SAPs/QAPPs are reviewed and approved through the Bay West peer review process, as appropriate. Adherence to properly documented field procedures will ensure that samples do not become contaminated through sampling activities and are representative of the site.

Sampling procedures outlined in the site SAPs/QAPPs are applied to field QC samples in the same way they are applied to regular field samples. Field QC sample containers must be labeled and transported, and the samples analyzed, in a manner identical to all other samples taken at a site.

All waste generated from sampling must be handled in accordance with project planning documents and appropriate regulations.

8.0 REFERENCES

Bay West, Inc., Bay West Chemical QA/QC Plan, CORP-MAN-005-64569V1

U.S. Environmental Protection Agency, Samplers Guide to the Contract Laboratory Program, EPA/540/P-90/006, December 1990.

Bay West

Standard Operating Procedure

Field Documentation

CORP-ENV-010-1578753

Revised: October 5, 2012

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Vice President of Operations
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Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

NOTE: This SOP is current as of the date printed on the bottom. Bay West personnel may produce paper copies of this procedure printed from the controlled-document electronic file located on the Intranet. However, it is their responsibility to ensure that they are trained and utilize the current version of this procedure.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

This Standard Operating Procedure (SOP) establishes the field documentation protocols required for all Bay West, Inc. (Bay West) field projects. Complete, accurate field documentation is required to track and monitor the progress of each project. Many clients require the field documentation to be included in the final deliverable. In addition, field documentation can be used to "reconstruct" field events: what went right and what went wrong that Bay West will use as lessons learned in its continuous improvement process. The field documentation requirements described in this SOP will pertain to Daily Diary Forms and a bound, dedicated site logbook, but may be used elsewhere as required.

1.2 Scope

This SOP applies to all personnel tasked to perform field documentation on all projects. The field documentation forms will include pertinent project information and may also include: boring logs, well installation logs, air, soil and/or groundwater sampling logs, and equipment lists, depending on the needs of the specific project. This information will be documented and the required forms provided in the site-specific plans. Information on documentation regarding sample custody and sample shipping may be found in Bay West SOP *CORP-ENV-004-1510208 Sample Custody* and Bay West

SOP *CORP-ENV-006-1510206 Packaging and Shipping of Environmental Samples*, respectively.

2.0 DEFINITIONS

Daily Diary Form – A preprinted Bay West form that once completed describes the details of what took place that day at the project site.

Bound Site Logbook – A bound, paginated book that is used to chronicle the activities of who-what-when-how regarding the daily site operations. Common names for these books are "Level Books" or "Rite-in-the Rain" logbooks.

3.0 RESPONSIBILITIES

Project Manager (PM) – Responsible for all aspects of the project and for ensuring field personnel compliance with the content of this SOP.

Site Supervisor – Responsible for ensuring use of proper and complete field documentation techniques during the performance of the field project.

Field Technicians/Field Samplers – Responsible for using consistent and appropriate field documentation techniques during the project and properly distributing/archiving the documentation at the conclusion of the project.

4.0 REQUIRED EQUIPMENT

This section provides a general list of equipment and supplies to be used but does not include all equipment specific to the job.

- Daily Diary form(with all applicable forms attached);
- Site-specific logbook(s);
- Permanent marking pen (a ballpoint or a Sharpie®)
- A pencil may be substituted for a permanent marker if heavy rain renders them useless. The information will require re-copying with a permanent marker in a dry environment.

5.0 PROCEDURES

Depending on the nature of the proposed activities and anticipated length of the project, the PM may require the use of either daily diary forms or a bound, dedicated site logbook for documentation purposes. Events/actions on site are to be documented as they occur. This practice will ensure the accurate depiction of the entire field effort.

Documented information should be factual and objective. Entries should include all daily activities, such as: site meetings, names of personnel entering and exiting the site, sampling data, weather conditions, any unusual occurrences, etc.

The PM is accountable to the client and therefore must possess a clear understanding of what actions were performed. To ensure Bay West's ability to accurately reproduce field activities, document the daily site activity with the following in mind:

- Do not simply record what you did; document why, how, who, where, and when it happened.
- What reasoning was behind your field approach?
- What methods did you use? Did anyone direct you to do it that way?
- Where did the field activities occur? What time did they occur?
- Were there any near misses on site?

Keeping the above questions in mind when documenting field activities will help to ensure the day's events are reproducible and have been well-documented.

6.0 DOCUMENTATION

The following sections describe common ways Bay West documents field activities. Due to the unique nature of our projects, not all scenarios can be listed here. The PM should determine the best way to document site activities.

6.1 Daily Diary Sheets

Daily diary sheets are to be completed by the Site Supervisor every day. Information will be documented in waterproof ink and kept current at all times. Documentation protocol will be the following:

1. Print Job Name, Job Number, Date, Project Manager, Names of Bay West Crew, and all other Personnel on-site.
2. Number the pages accordingly (if more than one page is required).
3. Provide a detailed description of the work performed. This written account should adhere to the general guidelines provided above.
4. Write the time for each entry in the left margin; use a 24 hour clock.
5. Place a single line through mistakes, initial and date each one.
6. Provide the full names, affiliations, and contact numbers for all persons noted as being on site.

7. Write late notes/entries as soon as possible and identify the entry as such. (Example: if something noteworthy occurred at 1000 and it is now 1500, simply document the activity in the next available space and note it as a late entry.)
8. Document any phone calls, including what was discussed and the instructions provided.
9. Document the location of the work performed and the selected level of PPE.
10. Draw site sketches, if appropriate.
11. Document the weather conditions.
12. Document the sampling and/or air monitoring methodology (if performed).
13. Document any waste generated and the volume created.
14. Note any change in conditions or deviations from the Work Plan (if any) and why the change was necessary.
15. Fill out the sample summary section, boring logs, etc. if appropriate.
16. Sign and date each sheet.

6.2 Site Logbooks

Site logbooks are to be completed by the appropriate site personnel. Site logbooks are to be bound and dedicated to one site. Entries for multiple sites will not be permitted in the same logbook. Information will be documented in waterproof ink and kept current at all times. Documentation protocol will be the following:

1. Print job name, job number, and volume number on the outside cover.
2. Print job name, job number, Project Manager name, and contact information on the inside cover.
3. Additionally, on the inside cover, write: "The Property of Bay West" and include the mailing address and phone numbers. Indicate "If Found, Please Call".
4. Number the pages in sequential order, if they are not already paginated.
5. Write the date on the first page of each new daily entry.
6. Provide a detailed description of the work performed/site observations. This written account should adhere to the general guidelines provided above.
7. Write the time for each entry in the left margin; use a 24-hour clock.
8. Place a single line through mistakes, initial and date each one.
9. Provide the full names, affiliations, and contact numbers for all persons on site.
10. Write late notes/entries as soon as possible and identify the entry as such (e.g., if something noteworthy occurred at 1000 and it is now 1500, simply document the activity in the next available space and note it as a late entry).
11. Document any phone calls, including what was discussed and the instruction provided.
12. Document the location of the work performed and the selected level of PPE.
13. Draw site sketches if appropriate. Draw a box around the site sketch to separate it from the text.
14. Document the weather conditions.
15. Document the number and type of samples collected, sample numbers, visual description, and laboratory analyses if appropriate.
16. Document any modifications in sampling locations and why.
17. Document the sampling and/or air monitoring methodology (if performed).
18. Document any photographs that were taken.
19. Document any waste generated including type, volume, and disposal location.
20. Note any change in conditions (if any) and why the change was necessary (e.g., stoppage of work due to inclement weather).
21. Initial the end of each daily entry.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

The site supervisor or team leader should complete the Daily Diary form or the site logbook on a daily basis. The site supervisor or team leader may delegate the role to another, qualified individual at the site, with the PM's concurrence.

8.0 REFERENCES

Bay West, Inc. Standard Operating Procedure, Sample Custody CORP-ENV-004-1510208, February 10, 2012

Bay West, Inc. Standard Operating Procedure, Packaging and Shipping of Environmental Samples, CORP-ENV-006-1510206, February 10, 2012

9.0 ATTACHMENT

Bay West Daily Diary Form

Bay West Inc.
Five Empire Drive
St. Paul, Minnesota 55103-1867

651/291-0456
FAX 651/291-0099
1-800-279-0456

DAILY DIARY

To be completed by Crew Leader *page* *of*

Job Name	**Job No.**	**Date**
Project Manager	**Bay West Crew**	
Personnel on Site ***(Client, Visitors, Bay West staff other than listed above)***		

Detailed description of work performed:

_____

Waste Generated:

_____

Change in Conditions (if any):

_____

_____

Sample Summary:

Samples Taken: ☐ Yes ☐ No	No. of Samples:	COC #:
Sample Destination: _____ Size and Type of Sample: _____		

Signature _____ *Date* _____

Bay West

Standard Operating Procedure

Field Equipment Decontamination

CORP-ENV-002-1578775

Revised: October 5, 2012

Review and Approval:

Developed by: _____ Date: October 5, 2012
Signature

QA/QC Manager
Title

Reviewed by: _____ Date: October 5, 2012
Signature

QA/QC Manager
Title

Approved by: _____ Date: October 5, 2012
Signature

Vice President of Operations
Title

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits,

changes, and revisions must be routed through the Document Management Coordinator.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

This Standard Operating Procedure (SOP) provides the procedures required for decontamination of field equipment. Decontamination of field equipment is required to ensure the safety of field personnel and the integrity of field samples by preventing cross contamination. Decontamination of field equipment reduces health hazards and prevents the spread of contaminants off site.

1.2 Scope

This SOP applies to all Bay West, Inc. (Bay West) personnel involved in field investigation activities when non-disposable field equipment is used. This equipment may include heavy equipment and sampling equipment.

2.0 DEFINITIONS

Contamination – Site-specific compounds/elements that can be transferred to other locations/sites or to personnel without the proper removal at the originating site.

Cross-contamination – The transfer of contamination — via equipment or personnel that have not been properly decontaminated — to less contaminated or uncontaminated samples or areas.

Decontaminated – Free of visible contamination and ready for use; when decontamination has been completed in accordance with this SOP.

Decontamination – The process of rinsing, scrubbing, or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross-contamination of samples or exposure to field personnel.

Equipment Blank (EB) – A sample collected and analyzed to check decontamination techniques and the sampling equipment for residual contamination. After the sampling equipment has been decontaminated, the EB is prepared by pouring deionized water (DI water) (usually provided by the laboratory) into the sample collection equipment and rinsing all internal surfaces that contact the sample. The EB is collected into the appropriate sample bottles for laboratory analysis and labeled as an “EB.” The site-specific plans will provide the rate of collection of EBs, the method, and type of water used.

3.0 RESPONSIBILITIES

Project Manager (PM) – Has overall responsibility of the project operations and ensures that decontamination operations follow the requirements provided in this SOP and the Accident Prevention Plan/Site Safety and Health Plan (APP/SSHP). If the analytical results for a target compound/element in the EB exceed one-half of the laboratory reporting limits (RLs) for a particular analyte, the PM must document the non-conformance and initiate a corrective action.

Site Supervisor – Manages site operations under the direction of the PM and communicates the requirements of the decontamination procedures presented in this SOP and the APP/SSHP to the field personnel. The Site Supervisor may also collect and document rinsate samples to provide quantitative verification that these decontamination procedures have been correctly implemented.

Field Personnel – Personnel that may perform equipment decontamination tasks at the site (which may include the Site Supervisor) and collecting the EB samples.

4.0 REQUIRED EQUIPMENT

This section provides a general list of equipment to be used for equipment decontamination. Additional apparatus may be required for site-specific equipment such as drill rigs and earthmoving machines:

- Gloves, safety glasses, and other protective clothing as specified in the APP/SSHP;
- Plastic sheeting;
- Stiff-bristle scrub brushes;
- Drain spade (for cleaning equipment tracks);
- Plastic buckets and/or troughs;
- Nalgene and/or Teflon wash bottles;
- Detergent (phosphate-free such as Alconox®, Liquinox®);
- Appropriate decontamination rinsing solutions such as tap water, pesticide grade hexane, methanol, DI water, etc. The required solutions will be delineated in the site-specific plans;
- 55-gallon drums or other containers to retain the decontamination fluids (sample container size is dependent on equipment being decontaminated);
- Disposable wipes or rags; and
- A high-pressure pump with soap dispenser or steam-spray unit may be required for larger equipment or particularly difficult contaminants such as viscous oils or tar-like substances.

5.0 PROCEDURES

All reusable equipment (non-dedicated equipment) used to collect, handle, or measure samples will be decontaminated before coming into contact with any sample. Decontamination of equipment will occur either at the central decontamination station or at portable decontamination stations set up at the work location, sampling location, drill sites, or monitoring well locations. If small decontaminated items are not immediately used, they will be covered with either plastic or aluminum foil to ensure cleanliness. If required, the centrally located decontamination station will include a separate pad on which the heavy equipment, drill rigs, and/or other large drilling equipment, such as auger flights, can be steam cleaned and properly stored to prevent cross-contamination.

The decontamination pad will be constructed in accordance with the site-specific plans to ensure contaminated water drains into a collection system. Collected water will be pumped into 55-gallon drums or portable tanks for storage, and if necessary, sent off site under a manifest by a licensed waste hauler, if required.

Decontamination fluids, such as solvents, may need to be segregated from other investigation derived wastes. Solvent disposal alternatives will be specified in the site-specific plans.

During any decontamination activity, personnel protective clothing and equipment as specified in the APP/SSHP will be worn. These may include: gloves, boots, safety glasses, face shields, respirators and other equipment.

5.1 Sampling Equipment Decontamination

- Soap used will be a non-phosphate detergent.
- Decontaminated equipment will be thoroughly rinsed with potable water or as specified in the site-specific plans.
- Decontaminated equipment will be allowed to air dry, if time allows, before being used.
- Procedures used for decontamination will be recorded in the site logbook.
- Sampling equipment that has come into contact with oily substances will be cleaned with methanol or other approved alternative solvent to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Specific requirements shall be followed as detailed in the site-specific plans.
- All solvents, if required, will be pesticide grade or higher purity and traceable to a specific source. The corresponding lot numbers will be recorded in the site logbook.
-

- Gloves, boots, safety glasses, face shields, and any other personnel protective clothing and equipment will be used as specified in the APP/SSHP.

5.2 Heavy Equipment Decontamination

Heavy equipment includes drill rigs and excavation equipment. The following specific steps are required when decontaminating this equipment.

Set up a decontamination pad that is large enough to fully contain the equipment to be cleaned. Use one or more layers of heavy plastic sheeting to cover the ground surface. With the equipment in place, spray areas of the equipment exposed to contaminated soils using a steam or a high-pressure sprayer. Be sure to spray down all surfaces that had the potential to contact contaminated soils.

1. Use drain spade, brushes, non-phosphate detergent and potable water or as specified in the site-specific plans to remove dirt whenever necessary.
2. If soapy water is used, rinse the equipment with clean, potable water or as specified in the site-specific plans. If using steam, the rinse step is not necessary if the steam does not contain a detergent. If the steam contains a detergent, this final clean water rinse is required.
3. Remove equipment from the decontamination pad and allow it to air dry, if time allows, before returning it to the work site.
4. Record equipment type, date, time, and method of decontamination in the appropriate logbook.
5. After decontamination activities are completed, collect all contaminated waste waters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles (as required). All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.3 Downhole Equipment Decontamination

Downhole equipment requiring decontamination includes: hollow-stem augers, drill pipes, casings, screens, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area should be set up to contain contaminated rinse waters and to minimize the spread of airborne spray. The decontamination area/pad requirements will be site-specific.
2. Set up a “clean” area upwind of the decontamination area to receive cleaned equipment for air drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed.
3. Place the object to be cleaned on plastic-covered sawhorses or other supports.

4. Using non-phosphate detergent and potable water in the high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and in gaps thoroughly. Use a brush, if necessary, to dislodge dirt.
5. If using soapy water, rinse the equipment using clean potable water or as specified in the site-specific plans. If using steam, the rinse step is not necessary if the steam does not contain a detergent. If the steam contains a detergent, this final clean water rinse is required.
6. Remove the equipment from the decontamination area and place in the clean area to air dry.
7. Record equipment type, date, time, and method of decontamination in the appropriate logbook.
8. After decontamination activities are completed, collect all contaminated waste waters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.4 Sampling Equipment Decontamination

Sampling equipment includes split spoons, spatulas, bowls, and other equipment used for sample homogenization that directly contact sample media. Follow these steps when decontaminating this equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line should progress from “dirty” to “clean” and end with an area for drying decontaminated equipment. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or the surfaces on which decontaminated equipment is to be placed.
2. Before washing, disassemble any items that might trap contaminants internally. Do not reassemble these items until decontamination is complete. Wash items thoroughly in a bucket of non-phosphate detergent and potable water or as specified in the site-specific plans. Use a stiff-bristle brush to dislodge any clinging dirt.
3. Rinse the item in potable water or as specified in the site-specific plans. Rinse water should be replaced as needed, generally when the water becomes cloudy.
4. If required by the site-specific plans, rinse the item with DI water, isopropyl alcohol, 10% nitric acid (for stainless steel, glass, plastic, and Teflon), or 1% nitric acid (for items made of low- carbon steel) followed by a distilled water rinse.
    a. **Note**: Care should be taken not to get nitric acid on skin or clothing. This step should not be used unless required by the sampling plan.

b. **Caution**: Do not allow nitric acid to contact methanol or hexane. Contain nitric acid waste separately from organic solvent waste.

5. If required by the sampling plan, rinse the item with methanol or another approved organic solvent.
6. If polar organic compounds such as pesticides, polychlorinated biphenyls (PCBs), and fuels are to be sampled, rinse the item with hexane or approved alternatives, followed by a second methanol rinse. This step should not be used unless required by the site-specific plan.
7. Triple rinse the item with water if solvents were used to clean the item, as required by the site-specific plans.
8. Shake off remaining water and allow the item to air dry completely, if time allows.
9. After drying, wrap the clean item in plastic wrap or in aluminum foil.
10. Record equipment type, date, time, and method of decontamination in the site logbook.
11. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable gloves, boots, and clothing. Place contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. (Refer to site-specific plans for waste management requirements).

5.5 Pump Decontamination

Follow these steps when decontaminating pumps:

1. Set up the decontamination area using plastic sheeting to cover the ground, tables, and other porous surfaces. Follow the site-specific plans for pump decontamination. For example, the plans may require setting up three containers in a triangle. One container at the base of the triangle would contain dilute (non-foaming) soapy water and the second container would hold potable water. The container at the apex would receive waste water. Place a container of DI water, if required, adjacent to the waste container on the same side as the potable water container.
2. The pump should be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first container. Place the discharge outlet in the waste container above the level of the waste water. Pump soapy water through the pump assembly until it discharges to the waste container.
3. Move the pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole-wetted parts must be immersed in the

potable water rinse. Pump potable water through the pump assembly until it runs clear.

4. If required, move the pump intake to the distilled water container. Pump distilled water through the pump assembly. Usually, three pump-and-line-assembly volumes will be required to thoroughly rinse the apparatus.
5. Decontaminate the discharge outlet by hand following the steps outlined in Section 5.4.
6. Place the decontaminated pump assembly in the “clean” area and allow it to air dry, if time allows. Intake and outlet orifices should be covered with aluminum foil to prevent the entry of airborne contaminants and particles.

5.6 Waste Disposal

Refer to site-specific plans for waste disposal requirements. The following are general guidelines for disposing of wastes:

- All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as a hazardous waste unless other arrangements are approved in advance.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions will be generated, it may be best to separate each type of waste in a separate container. This may permit the disposal of wash water and rinse water in a sanitary sewage treatment plant rather than as a hazardous waste.
- If an industrial waste water treatment plant is available on-site, the disposal of acid solutions and solvent-water solutions may be permitted there. The site-specific plans will indicate the proper disposal methods.
- Unless required by the site specific plans, plastic sheeting and disposable protective clothing may be treated as a solid, nonhazardous waste.

6.0 DOCUMENTATION

All notes/comments associated with the decontamination of field equipment will be recorded in a project-specific field notebook in accordance with the Bay West SOP for Field Documentation, CORP-ENV-010-1578753.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

Whenever possible, sampling is performed in a clean to dirty progression. Equipment decontamination is performed between each sample collection location. Depending on

project requirements, an EB may be collected. When possible, EBs are collected after "dirty" samples to assess the "worst case" decontamination effectiveness. Successful decontamination will result in an EB with no detections above laboratory RLs. If laboratory results for an EB exceed the RLs, the PM must document this and initiate a corrective action.

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics, respectively. These steps should not be used unless specifically required by the site-specific plans because of potential acid burns and ignitability hazards.

If the field equipment rinsed with solvents is not allowed to air dry properly before use, volatile organic residue may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted; volatilization is rapid in the summer, but slow in the winter. Approved decontamination solvents are listed in the site-specific plans.

8.0 REFERENCES

Bay West, Inc. Standard Operating Procedure for Field Documentation, CORP-ENV-010-1578753.

Department of Energy, Hazardous Waste Remedial Actions Program, *Standard Operating Procedures for Site Characterization,* DOE/HWP-100, July 1990.

Department of Energy, Hazardous Waste Remedial Actions Program, *Quality Control Requirements for Field Methods,* DOE/HWP-69/RI.

American Society for Testing and Materials, *Standard Practice for Decontamination of Field Equipment at Nonradioactive Waste Sites,* ASTM 05088-90, June 29, 1990.

U.S. Environmental Protection Agency, Region II, *CERCLA Quality Assurance Manual,* Revision 1, 1989.

U.S. Environmental Protection Agency, Region IV, *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual,* 1986.

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods,* EPA/540/Procedures-87/001.1, 1987.

Bay West

Standard Operating Procedure

Packaging and Shipping of Environmental Samples

CORP-ENV-006-1510206

Revised: February 10, 2012

Review and Approval:

Developed by: John D. Verlan Date: 02-15-2012
Signature
QA/QC Manager
Title

Reviewed by: John D. Verlan Date: 02-15-2012
Signature
QA/QC Manager
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Approved by: Date: 02-15-2012
Signature
Vice President of Operations
Title

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

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1.0 INTRODUCTION

1.1 Purpose

The purpose of this Standard Operating Procedure (SOP) is to establish packaging and shipping requirements and guidelines for environmental sample shipping. Proper packaging and shipping is necessary to ensure the protection and the integrity of environmental samples shipped to the laboratory for analysis.

2.0 DEFINITIONS

Environmental Sample - Environmental Sample is any sample that has less than reportable quantities for any hazardous constituents according to Department of Transportation (DOT) regulations for promulgated in 49 CFR - Part 172.

Chain-of-Custody – Chain-of-Custody is a legal term that refers to the ability to guarantee the identity and integrity of the sample (or data) from collection through reporting of the test results.

3.0 RESPONSIBILITIES

Sampler – The sampler is required to maintain Chain-of-Custody of the samples at all times. Only people associated with the project may handle the samples. The duties of the sampler include: proper labeling of the samples, completion of the Chain-of-Custody form, proper packing of the shipping container, most often a cooler, and transportation of the shipping container to the courier meeting point or to an overnight delivery service. The Chain-of-Custody always accompanies the samples at all times.

Site Supervisor - The site supervisor is responsible for ensuring that the Chain-of-Custody is maintained, packaging and sampling procedures are conducted in accordance with this SOP and the site specific SAP. The site supervisor is also responsible for ensuring that the proper laboratory analysis of the samples is requested by Bay West.

Project Manager - The Project Manager is responsible for maintaining logbooks and forms, tracking the Chain-of-Custody and for approving and documenting techniques not specifically described in this SOP.

4.0 REQUIRED EQUIPMENT

This section provides a list of equipment to be used but does not necessarily include all equipment such as sample containers and personal protection equipment. The following is a general list of equipment that should be obtained prior to initiating field work:

- Coolers with return address labels of the Bay West office;
- Heavy-duty plastic garbage bags;
- Plastic zip-top bags, small and large;
- Clear tape;
- Fiber tape;
- Duct tape;
- Bubble wrap;
- Ice;
- Chain-of-Custody seals;
- Indelible black ink pens and markers;
- Completed Chain-of-Custody record or CLP custody records, if applicable;
- Completed Bill of Lading (if required); and
- "This End Up" and directional arrow labels;
- Saturday Delivery Labels (if required).

5.0 PROCEDURES

The following steps must be followed when packing sample bottles and jars for shipment:

1. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape. Line the cooler with a large heavy-duty plastic garbage bag;
2. Be sure the caps on all bottles are tight (will not leak). Check to see that sample labels and chain-of-custody records are completed properly and they match *exactly*;
3. Place all bottles in separate and appropriately-sized, plastic zip-top bags, remove air pockets and close the bags. Up to three VOA vials may be packed in one bag. Glass containers must be wrapped in bubble wrap prior to insertion into the bag. Optionally, place bagged and bubble-wrapped VOA vials in a quart/gallon metal can and then fill the can with additional bubble wrap;
4. Place a sufficient layer of bubble wrap on the bottom of the cooler and then place the bottles and cans in the bag with sufficient space to allow for the addition of more bubble wrap between the bottles and cans. It is mandatory to place glass sample bottles and jars into the cooler *vertically*. Due to the strength properties of a glass container, there is significantly less chance of breakage when the container is packed vertically rather than horizontally;

5. Put ice in large, plastic, zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place these ice bags on top of or between the samples. Several bags of ice are required for temperature control. Fill all remaining space between the bottles or cans with bubble wrap. Securely fasten the top of the large garbage bag inside the cooler with fiber or duct tape;
6. Place the completed Chain-of-Custody Record or the Contractor Laboratory Program (CLP) Traffic Report Form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler's lid, and then close the cooler. The sampler keeps one copy of the Chain-of-Custody Record. Include any additional paperwork sent to notify the laboratory of project information (laboratory notification checklist) or if a sample is suspected of containing any substance for which laboratory personnel should take additional safety precautions;
7. Fiber tape or duct tape shall be wrapped around each end of the cooler two times and completed custody seals affixed to the top opposite sides of the cooler, half on the fiber tape or duct tape so that the cooler cannot be opened without breaking the seal. Complete an additional two wraps with fiber tape or duct tape; place clear tape over custody seals; and
8. The shipping container lid must be marked "THIS END UP" and arrow labels which indicate the proper upward position of the container should be affixed to the cooler. A label containing the name and address of the shipper (Bay West) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo-only Aircraft, Flammable Solids, etc.) are not permitted to be on the outside of the container used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container; or, when shipping by common courier, the airbill or the Bill of Lading shall be completed and attached to the lid of the shipping container.

6.0 DOCUMENTATION

All notes/comments associated with the packaging and shipping will be recorded in a project specific field notebook in accordance with this Bay West. A copy of the Chain-of-Custody will be sent to the Project Manager and entered into the Bay West Document Management System. The copy of the original Chain-of-Custody retained in the field will be sent to the project file.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

Each sample cooler must be packaged and preserved with sufficient ice in order for the laboratory to receive the samples intact and at the proper temperature, 4°C ± 2°C. If there are additional preservation requirements, e.g. Dry Ice, these will be presented in the site specific SAP.

Samples shall be wrapped and packaged to prevent cross contamination. Trip blank samples are provided by the laboratory for analysis to assess potential cross contamination.

Temperature Blank samples may be provided by the laboratory. These plastic bottles should remain in the bottom of the cooler. Do not wrap them in bubble wrap. If no temperature blanks are present upon receipt of the cooler(s), the lab will use an IR Temperature Gun to determine the sample's receipt temperature.

7.1 Restrictions/Limitations

Holding times for the sample analyses must not be exceeded. It is recommended that samples be packed in time to be shipped nightly for overnight delivery. If not, the samples should be shipped the next morning. Use caution when shipping samples for Saturday delivery; make arrangements by calling the laboratory before sending the samples.

8.0 REFERENCES

U.S. Environmental Protection Agency, *Sampler's Guide to the Contract Laboratory Program*, EPA 540-R-07-06, July 2007.

U.S. Environmental Protection Agency, Region IV, Field Sampling Quality Control, SESD PROC-01-R3, October 2010.

U.S. Army Corps of Engineer, EM 200-1-3, *Requirements for the Preparation of Sampling and Analysis Plans*, February 2001.

Air Monitoring Instrumentation Manual

CORP-MAN-007-52523V4

Revised: April 11, 2007

Review and Approval:

Revised by: _____
Signature

Corporate Health and Safety Manager Date: April 11, 2007
Title

Reviewed by: _____
Signature

Corporate Quality Control Manager Date: April 11, 2007
Title

Approved by: _____
Signature

Vice President of Operations Date: April 11, 2007
Title

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations, Corporate Health and Safety Manager or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

Preface

Air monitoring instruments are used nearly every day at Bay West to make decisions regarding health and safety risks, hazardous waste characterization, and to track and identify contamination sources. This training manual is designed to assist the field technicians, site supervisors, and project managers in understanding how to operate Bay West's air monitoring instruments. This manual includes condensed instructions for instruments which detect organic and inorganic vapors and gases; oxygen deficiency and excess, and the presence of combustible gases.

Air monitoring is performed for different reasons. The purpose of monitoring will vary with the situation as well as the actual, potential, or anticipated hazard. Active monitoring is an integral part of many of Bay West's Health and Safety Plans (HASPs) and in many cases, required by law. A well-written HASP for hazardous materials sites will discuss the specific air monitoring requirements in detail.

This manual is not designed to be all inclusive of every instrument in use at Bay West facilities or field locations. It is designed to be a supplement to the manufacturer's operator manual. We also do not suggest that individuals using this manual replace trained health and safety professionals. It is always best to consult a trained professional when using air monitoring instrumentation as part of a health and safety program. A lack of basic understanding of how monitoring instruments operate and their inherent limitations can result in misinterpretation of collected information, which may jeopardize the health and safety of all concerned. Please familiarize yourself with the contents of this manual before using the air monitoring equipment. If in doubt, please ask for help from the Corporate Health and Safety Manager or other staff knowledgeable with instrumentation use.

BE AWARE OF CHEMICALS OR COMPOUNDS THAT ARE SPECIFICALLY DESCRIBED IN THE OPERATIONS MANUAL THAT CANNOT BE DETECTED BY THE INSTRUMENT. For example, although the ionization potential (IP) for formaldehyde is 10.88 eV, well within the range of the TVA 1000 FID instrument (a formaldehyde response or correction factor is listed in the FID manual), the manual specifically states that the FID does NOT register formaldehyde (due to its inorganic properties).

THIS MANUAL PROVIDES ONLY A CONDENSED SUMMARY VERSION OF OPERATIONS AND CALIBRATION. PLEASE CONSULT THE USER MANUAL FOR THE INSTRUMENT FOR DETAILED OPERATION INSTRUCTIONS.

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1.0 Instructions for the Use of the Dräger Detector Tubes and Hand Pump - Tool Watch #2650-03 to 06

1.1 Introduction

Chemical specific air-sampling tubes are used to determine specific air concentrations of a contaminant. They are often used to determine respiratory upgrade and downgrade parameters. For example, using a Dräger mercury vapor detector can to determine respiratory requirements for spill clean up.

Things to remember when using indicator tubes: select the correct box of tubes (appropriate chemical name and appropriate concentration range), read the directions that accompany the box of tubes before attempting to take a sample, replace directions in the box of tubes, and check the expiration date on the box of the tubes. Do not use the detector tubes if expired. In a crunch, outdated tubes could be used for confirming identity -- **not for quantitation. NOTE**: Dräger tube results are only accurate in the $\pm$ 15-20% range under the best conditions, additional analysis methods to confirm the Dräger results should be used whenever available.

1.2 Instructions for Use

1.2.1 Short-Term Tubes and Pump

- Check the bellows for leakage. Depress the bellows without a tube attached. Once depressed attach an unopened tube. Release the pump bellows, the pump should not open. If the bellows moves significantly after 15 minutes the valves need to be changed. Contact the health and safety specialist for assistance.
- Use the tubes within the recommended temperature range. Again, at low temperatures, the tubes could be used for confirming identity -- **not for quantification.**
- Open both of the tube tips with the Dräger tube opener or alternatively the round hole in a key.
- Insert the front tip opening into the pump, the arrow must point towards the pump
- Once the pump and tube assembly has been prepared, advise entry personnel of the number of strokes/pumps and the color change to expect. There is a stroke counter on the hand pump.
- Note the interfering chemicals listed in the tube directions. Sometimes the only way to detect and measure a chemical is by using the interfering agent as a surrogate.

1.2.2 Long-Term Detector Tubes

These tubes are used for long-term employee exposure monitoring and the hand pump is not used. Please refer to the instructions in the tube kit for more details. Basic instructions for use are:

- Select the appropriate detector tube for the contaminant of interest.
- Use the tubing holder the break off the front end of the detector tube. (Both ends of this tube are not to be broken!)

- Once the tube is broken, insert the tube (broken end up) in the holder and hang the tube and holder in the employee's immediate breathing zone.
- After the appropriate sampling time (typically 4-8 hrs) observe the tube for the color change mentioned in the specific tube directions. The tubes read exposures directly in ppm/hrs
- Do not use the diffusion tubes for less than 1 hour exposure time as it will yield inaccurate results.

Manage discarded tubes properly. Read the instruction sheet. Some tubes are hazardous because they contain, for example, fuming sulfuric acid. These tubes should be managed as a hazardous waste. Other tubes do not contain hazardous material and can be discarded in the solid waste stream. Take care to prevent cuts and punctures when handling opened tubes.

1.3 Documentation

Air borne chemical concentrations will need to be documented as part of Bay West specific project Health and Safety Plans (HASP). Employee exposure records become part of the permanent medical file and must be documented by the Health and Safety Specialist.

2.0 Quick Start-Up Instructions for Use of the PE Photo Vac 2020 - Tool Watch #200020

2.1 Instructions for Use

1. Attach Probe

2. Press ON/OFF Key

3. Meter should be initially set in the AUTO function with 1 second updates

4. Meter displays the time, date and peak ppm value.

5. Calibrated for 100 ppm isobutylene, response factor of 1.0. For use with other organic compounds see the response factors listed in the instrument manual Section 8.7, "Table of Response Factors" pages 97-99. The following calculation can be used:

Response Factor = Actual Concentration in PPM/Response of Instrument

NOTE: The data logger will eventually fill up with data and make further measurements impossible. Need to switch to MANUAL mode to clear the memory of sample data points. For details talk to one of the Health and Safety Specialists.

INSTRUCTIONS CONTINUED ON NEXT PAGE

2020 Photoionization Detector Operational Reference Guide

INTRODUCTION

This TechTIP is a reference guide for day to day operation of the 2020 Photoionization Air Monitor. For more detailed information, please refer to the 2020 User's Manual.

The 2020 measures, displays and datalogs the total concentration of Volatile Organic Compounds (VOCs). The 2020 is a non-specific instrument, therefore it does not distinguish between individual compounds. The reading displayed represents the total concentration of all VOCs present in the sample. The 2020 displays concentration in ppm.

The 2020 operates automatically. The display updates itself once per second. The 2020 is always performing short term exposure limit (STEL), time weighted average (TWA), and PEAK calculations.

CALIBRATION

The 2020 can be calibrated for a particular compound of interest if the environment contains a single known Volatile Organic Compound. Please refer to the **Programming Calibration Memories and Response Factors** section.

If the environment monitored is complex, containing different VOCs, or contains VOCs with unknown Response Factors, a Response Factor equal to 1 should be used. This can be achieved performing the **Standard Calibration** outlined below. The default Response Factor is equal to 1.0.

Standard Calibration

- → Press the Instrument ***On/Off*** Key to turn on the 2020. The EPROM software version will be displayed.
- → Select ***Enter*** to access the menu.
- → Select ***Set***
- → Select ***Cal*** for Calibration
- → Select ***Zero*** to zero the instrument. This process will take 60-90 seconds.

Note: Zeroing can be done in ambient air if the environment is free of VOCs. Use a cylinder of ultra zero air if the ambient air has VOCs present.

- → Select ***Span***. The span gas is 100 PPM isobutylene. The process will take 1-2 minutes to equilibrate.

Calibration is now complete and the 2020 is now ready to sample.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Programming Calibration Memories and Response Factors

The 2020 has 15 Cal Memories and can be calibrated with 15 different span gases or response factors if desired.

If you will be calibrating directly from the portable cylinder, connect a flow- match regulator (Photovac Part No. MX350006) to each tank. You must use a separate regulator for each compound to prevent cross contamination.

If you are using Tedlar™ gas bags, prepare the bags of calibration gas. Use a different gas bag and a gas bag adapter for each concentration and for each type of calibration gas. You can use the same gas bag to zero all the Cal Memories, however you must refill the bag before zeroing each Cal Memory.

To program the Cal Memories:

- Press the Instrument ***On/Off*** Key to turn on the 2020. The EPROM software version will be displayed.
- Select ***Set***
- Select ***Cal*** for Calibration
- Select ***Mem***
- Select the desired Cal Memory with the ***Next*** and ***Prev*** keys.
- Press ***Chng*** to change the parameters of the Cal Memory. Select ***User*** or ***Lib***
- If you selected ***User*** enter the name for the Calibration memory. Press the Enter key and the 2020 will then prompt you to enter the response factor (RRF). You will next be prompted to enter the alarm levels for each mode.
- If you entered ***Lib*** use the ***Next*** and ***Prev*** keys to select the required library. Please note that the response factor (RRF) is preprogrammed into the library.
- Calibrate the instrument , and when the calibration is completed, the calibration information is automatically stored in the selected Cal Memory.
- Repeat the procedure for each Memory you need.

INSTRUCTIONS CONTINUED ON NEXT PAGE

2020 Photoionization Detector Response Factors (10.6 eV)

Compound	Response Factor	Compound	Response Factor
Acetaldehyde	10.5	Ethanol	8.8
Acetone	1.2	Ethyl Acetate	3.8
Acetophenone	2.0	Ethyl Acrylate	2.3
Acrolein (2-Propenal)	4.0	Ethylbenzene	0.5*
Acrylic Acid	10.9	Ethyl Cellosolve (2-Ethoxyethanol)	1.3*
Acrylonitrile	ND	Ethylene Glycol	0.1
Allyl Chloride (3-Chloro-1-Propene)	3.9	Ethyl Ether (Diethyl Ether)	1.2*
Benzene	0.5	Ethyl Mercaptan (Ethanethiol)	0.6
Bromoform (Tribromomethane)	2.0*	Ethylene	10.1
1,3-Butadiene	0.7	n-Heptane	2.4*
n-Butane	0.2	n-Hexane	4.7
n-Butanol	3.4	2-Heptanone	2.1
n-Butyl Acetate	2.3	Hydrogen Sulfide	3.3
Butyl Acrylate	1.8	Isoamyl Acetate	1.8*
Butyl Cellosolve	3.1	Isobutyl Acetate	2.6
n-Butyl Acrylate	1.8	Isobutyl Alcohol	0.3
n-Butyl Mercaptan (Butanethiol)	0.6	Isobutyraldehyde	1.1
Carbon Disulfide	1.3	Isopentane	8.2
Chlorobenzene	0.4	Isoprene (2-Methyl-1,3-Butadiene)	0.6
Crotonaldehyde (2-Butenal)	1.2*	Isopropanol	4.4
Cumene (Isopropyl Benzene)	0.6*	Isopropyl Acetate	2.6
Cyclohexane	1.3	Isopropyl Ether	0.8*
Cyclohexanol	3.4	Mercaptopropionic Acid	0.1
Cyclohexanone	0.9*	Methacrylic Acid	000
Diacetone alcohol	1.8	Methyl n-Amyl Ketone (2-heptanone)	2.1
1,2-Dichlorobenzene (ortho-)	0.5*	Methyl Bromide (Bromomethane)	1.6
Ethyl Acrylate	0.9	Methyl tert-Butyl Ether (MTBE)	0.8
cis-1,2-Dichloroethylene	0.8	Methyl Chloroform (1,1,1 –TCA)	000
trans-1,2-Dichloroethylene	0.4	Methylene Chloride	000
N,N-Dimethylformamide (DMF)	0.8*	Methyl Ethyl Ketone (2-Butanone)	0.8
1,4-Dioxane	1.3	Methyl Isobutyl Ketone (MIBK)	1.0*
Epichlorohydrin	6.5*	Methyl Mercaptan	0.5

INSTRUCTIONS CONTINUED ON NEXT PAGE

2020 Photoionization Detector Response Factors (10.6eV) Continued

Compound	Response Factor	Compound	Response Factor
Methyl Methacrylate	1.4*	Therminol	000
Monomethylamine	1.3	Toluene	0.5
n-Nonane	1.4*	1,1,1 – Trichloroethane	000
iso-Octane (2,2,4-Trimethylpentane)	1.2*	Trichloroethylene (TCE)	0.5
n-Pentane	10.4	1,1,1 – Trichloroethane	000
Phenol	1.1	Triethanolamine	ND
Poylpropylene	0.8	Trimethylamine	0.9
Propane	000	1,2,4-Trimethyl Benzene	2.3
n-Propanol	5.1	1,3,5- Trimethyl Benzene	1.7
Propionaldehyde (Propanal)	14.8	Vinyl Acetate	1.2*
n-Propyl Acetate	3.1	Vinyl Bromide	0.4
Propylene	1.2	Vinyl Chloride (Chloroethylene)	1.7
Propylene Oxide	5.8	Vinylidene Chloride (1,1-DCE)	0.8
Styrene	0.4*	ortho-Xylene	0.5*
Syltherm XLT	9.8	meta-Xylene	0.5*
Tetrachloroethylene (Perchloroethylene)	0.5	para-Xylene	0.5*
Tetrahydrofuran	1.5		

The *Photovac 2020* compound Response Factors were determined over the range 0 - 500 PPM, based on a 100 PPM Isobutylene calibration. Isobutylene RF = 1.0. The following formula was used for calculation of Response Factors:

$$\text{Response Factor} = \frac{\text{Actual Concentration}}{\textit{2020}\ \text{Response}}$$

A Response Factor less than 1.0 indicates a compound response better than that of Isobutylene. A Response Factor greater than 1.0 indicates a lower response than that of Isobutylene.

The following Response Factors are pre-programmed into the memory of the *Photovac 2020*. There is space in the *2020* for up to 15 user-entered Response Factors. **Unless otherwise noted, when using Response Factors, results are expected to be accurate to +/- 10 PPM or +/- 25%, whichever is greater.**

Standards used for determination of *Photovac 2020* Response Factors were from certified gas cylinders, +/- 2% analytical accuracy.

* Response Factors for these compounds were determined over the range of 25-250 PPM. Standards were prepared by addition of neat liquid to Ultra Zero Air. When using Response Factors for these compounds, results are expected to be accurate to +/- 15 PPM or +/- 35% whichever is greater.

For further information contact your area representative or Photovac, Inc.

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3.0 Instructions for the Use and Calibration of the MSA Escort Elf Personal Sampling Pumps - Tool Watch # 215010-012

3.1 Introduction

The MSA Escort Elf pump is an air sampling device most frequently used for the collection of particulate air contaminants encountered while working. The collection of samples is used primarily for the purpose of documenting occupational exposure over the course of a working day and the life of a project. The pumps are light weight and meant to be attached to the wearer's belt or waist with sample collection occurring via a small tube affixed near the wearer's collar/neck (breathing zone). Selective filter cassettes are used and serve as the media from which the contaminant is desorbed and analyzed by a laboratory. Below is an overview of the pump's operating specifications.

Section 1, General Information

Operating Specifications	
ELECTRICAL CHARACTERISTICS	
POWER SUPPLY	4.8-volt battery pack of four nickel-cadmium cells
BATTERY PACK CAPACITY	1.8 ampere hour
BATTERY PACK RECHARGE TIME	14-16 hours (overnight) with recommended chargers
CHARGING TEMPERATURE	10° to 30°C (50° to 86°F)
TYPICAL BATTERY PACK LIFE	300 or more charging cycles
OPERATING AND PHYSICAL CHARACTERISTICS	
FLOW CONTROL	Volumetric flow rate is controlled at the set-point with automatic compensation for battery voltage, altitude, temperature, and sample load changes
OPERATING RANGE	0.5 TO 3.0 LPM
	5 to 30 inches water load up to 1.0 LPM; up to 30 inches of water load up to 2.0 LPM; up to 20 inches of water load up to 2.5 LPM; and up to 10 inches of water load up to 3.0 LPM
FLOW BLOCKAGE DETECTION	When flow is blocked the FLOW FAULT LED indicator lights and stays lighted until the cause of flow blockage is removed. If blocked for longer than 1.5 minutes, the pump shuts down. Pumps that are ordered for coal mine dust sampling are factory-programmed to *not* shut down
FLOW READOUT	Liquid crystal digital display with 0.01 LPM resolution from 0.5 to 3.0 LPM
	Accuracy ±2.5% from 1.0 to 3.0 LPM, 0° to 45°C ±5% from 1.0 to 3.0 LPM, -20° to 0°C and 45° to 65°C ±5% from 0.5 to 0.9 LPM, 0° to 45°C.

3.2 Start-up/Operation

The following information has been copied from the operation manual. Additional detail and information can be found in the manual.

LED Indicator Operation

- The RUN/HOLD LED assures the user that the unit is operating, even in poorly lit, noisy environments.
- The RUN/HOLD LED also indicates the HOLD function. If the pump is switched to HOLD, the pump shuts down, the timer stops, and the LED is switched to a blinking mode. When the RUN/HOLD switch is pressed again, the pump will restart and the timer will continue from its "hold" value.
- The LOW BATT LED indicates operation of the low battery detection circuit. When the battery pack drops to 4.1 volts, the circuit causes the pump to shut down, the timer to stop, and the LOW BATT LED to light. This limits further battery drain to just a few milliamperes, which, in turn, helps prevent deep discharging and battery pack damage upon recharging. It also helps prevent the invalidation of the sample being taken, because the timer stores the actual sample time and the flow rate is controlled up to the time of shut-down.
- The FLOW FAULT LED indicates operation of the flow blockage detection circuit. If the sample inlet (or the sampling device connected to the pump) is blocked, the FLOW FAULT LED lights until the cause of the flow fault is removed.
- The pump is normally factory-programmed so that a fault longer than 1.5 minutes will shut down the pump. The pump may be restarted from a flow fault shutdown by pressing the RUN/ HOLD switch. Restarting in this manner allows continuation of the sample period without having the timer reset to zero.
- The Flow Fault LED is lighted whenever the pump flow rate is more than ±0.1 LPM different from the set-point. It is normal for it to be lighted briefly while the pump is started or when a new set-point is selected.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Operating the Pump

1. Charge the battery pack for 16 hours using the MSA Omega (Ω) Charger.
   - Read the WARNING on the battery pack.
2. Connect the desired sampling device to the inlet fitting.
3. Press ON/OFF button until the RUN/HOLD LED lights.
   - The Flow Fault and Low Bat LEDs will then light. During the time these LEDs are being lighted, a number from 0 to 999 will be displayed. This is the total of the pump's operating hours since its last calibration.
   - When all three LEDs are lighted, the display changes to **8.88 LPM/MINS**, to show all segments are functional.
   - The pump will then begin operation. The LOW BAT LED will be dark if the battery voltage is above 4.1 volts. The Flow Fault LED will be dark when the flow rate is within 0.1 LPM of the setpoint.
4. Use the ↑ and ↓ switch buttons to adjust the flow setpoint if a new flow rate is desired. When either button is pressed:
   - RUN/HOLD LED will be dark
   - Display will show the flow setpoint being stepped up or stepped down to the new setpoint.
5. The RUN/HOLD button may be pressed to interrupt sampling without causing the timer to reset to zero.
6. Press the ON/OFF button at the end of sampling.
   - Total sample minutes will be on the display and will remain until the pump is turned ON again.
7. Check calibration monthly (every 200 hours of use for coal mine dust sampling).
8. Operate the pump with a sampling device connected to the inlet. Operation without a sampler attached will reduce the life of the pump inlet filter. Operation at very low flow rates may be unstable without an attached sampling device, such as a charcoal tube.

INSTRUCTIONS CONTINUED ON NEXT PAGE

3.3 Calibration Steps

1. Check pump inlet filter for dust; replace if heavily loaded.
2. Turn ON the pump and allow it to reach its flow rate setting.
3. If the pump is not set for 2.50 LPM, set it to 2.50.
4. Allow the pump to operate for 10 to 15 minutes at 2.50 LPM. To check for leaks, temporarily block the pump inlet; 0.02 LPM or less should be displayed. If not, check the inlet filter cover and o-ring for correct assembly.
5. Leave the pump set for 2.50 LPM and turn OFF the pump.
6. Connect a primary calibration standard to the pump.
7. Turn ON the pump; as the pump goes through its self-check sequence, press and hold the ON/OFF and RUN/HOLD switches simultaneously until the display reads CAL. Immediately release both buttons.

   NOTE: Failure to release both buttons will turn OFF the pump.

   - The display will show CAL and a countdown sequence of numbers from 9 through 0; thereafter, it will alternately display CAL and 2.50 LPM.
8. Operate the calibration standard and obtain at least six readings which are very close to one another.
9. Use the ↑ and ↓ switch buttons to step the pump display up or down until it agrees exactly with the average of the six readings.
10. Turn OFF the pump; the ELF sensor is now calibrated. Repeat monthly or every 200 hours of use.
11. When you next turn ON the pump, the operating hours will be reset to **0** and be displayed during the lighting of the FLOW FAULT and LOW BAT LEDs.

3.4 Documentation

All calibration results, repairs and maintenance must be documented in the instruments log book. The book should be inside the instrument's carrying case. This provision is required for some government contracts and is good industry practice. If the logbook is missing, please contact one of the health and safety specialists for a replacement.

4.0 Instructions for the Use and Calibration of the HNU Photo-Ionizer - Tool Watch #200011

4.1 Introduction

The HNU photoionizer is a multifunction instrument capable of measuring trace levels of organic contaminants in air including: chlorinated hydrocarbons, heterocyclics, aromatics, aldehydes and ketones as well as several inorganic gases with a ionizing potentials. This instrument can be used for plume spread at spill sites, and headspace analysis of soil, water and other matrices'.

4.2 Operation

4.2.1 Start-Up

Attach the probe to the readout module with the 12-pin connector. Do not force the connection or damage to the pins could result. Once the plug is inserted, turn the connector ring clockwise until a distinct click is heard or felt.

Turn the function switch to the BATT position. The meter needle should deflect to the blue area at the right hand side of the meter scale plate. The fan should be operating, and a slight humming can be heard. Look briefly directly into the probe inlet and observe a lamp glow (purple light). Since prolonged UV light can cause eye damage, it is desirable to wear tinted safety glasses when this lamp check is performed.

Turn the function switch to the STANDBY position. The fan will continue to operate but the lamp is of. The meter needle moves to the left out of the blue area on the meter scale. Adjust the zero knob until the needle rests at zero (0). The meter can only be electronically zeroed while in the STANDBY mode.

Check the span setting for the lamp being used and adjust as necessary using the span control knob. The correct span settings are:

Lamp Voltage (eV)	Span Setting
9.5	1.0
10.2	9.8
11.7	5.0

4.2.2 Calibration

For calibration, a standard calibration span gas is used. The HNU calibration gas consists of approximately 100 ppm isobutylene in air, referenced to benzene. The regulator should be connected to the calibration gas canister and the outlet connected to the HNU probe via flexible tubing.

Turn the function switch to the 0-2000 range position (X100). Open the valve on the regulator and allow gas to enter the probe. The meter should read very close to the ppm concentration listed on the reference gas tank label. Adjust the span pot slightly until an exact reading is obtained.

Turn the function switch to the 0-20 position. The needle reading should deflect full scale to the right.

Return the function switch to the 0-200 position. The needle should once again indicate an accurate reading of the span gas concentration.

Turn the function switch to the OFF position. Close the valve on the calibration gas tank regulator and disconnect it from the probe inlet. The HNU (PI-101) is now ready for field use.

4.2.3 Battery Charging

Turn the function switch to OFF position. The probe must be attached in order for the unit to charge. Plug the miniplug into the charger jack labeled for that purpose. Connect the power cord to the AC source as the last step to avoid any electrical damage to the unit. The LED on the charger unit should be on. Charge the unit for 8-10 hours.

4.3 Documentation

Calibration, maintenance and repair records must be documented. This is required in a number of federal contracts and is a good industry practice. The HNU log book is stored in the instrument carrying case. If the log book is missing contact one of the safety and health specialists for a replacement.

5.0 Calibration and Use of the thermo TVA 1000 PID/FID - Tool Watch #200121-123

TVA-1000B
Toxic Vapor Analyzer
Operation, Configuration, and Maintenance

Style AA

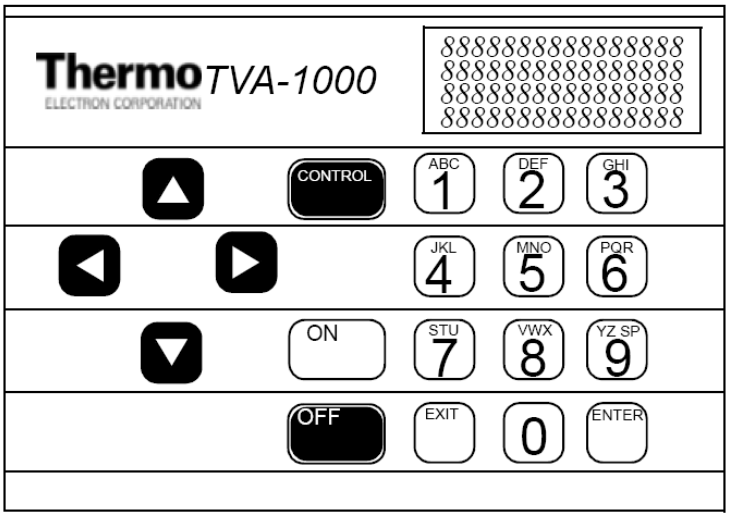

Figure 6. Keypad

NOTE: To activate OFF, CONTROL, EXIT, and ENTER functions, press and HOLD the key for approximately 1/2 second.

Key	Function
ON	The ON key enables power from the battery to the instrument.
OFF	The OFF key disables power from the battery to the instrument.
CONTROL	The CONTROL key is multi-function and is used to turn the pump, PID, and FID on or off, and to ignite the FID.
EXIT	The EXIT key clears any entry made in error or bypasses information that you do not want to change, and clears error or warning screens.
ENTER	The ENTER key has three functions: 1. Press ENTER if you have typed one or more characters and wish to keep that information. 2. Press ENTER to respond to a menu question. 3. Press ENTER instead of the LOG key on the standard probe to initiate logging.
Left/Right Arrows	The left and right arrow keys move character entry positions.
Up/Down Arrows	The up and down arrow keys make page selections or scroll through options in SETUP entry screens.

INSTRUCTIONS CONTINUED ON NEXT PAGE

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Key	Function
Alphanumeric	The alphanumeric keys enable you to type letters or numbers into various menus. If a display asks for a number only, simply press the desired key. Two steps are required to type an alphanumeric character. First, press the key with the desired letter or number. The screen then displays a selection prompt at the bottom in which 1 = first letter, 2 = second letter, 3 = third letter, and 0 = number. Press the appropriate key to execute the selection. Three uses: ♦ Select menu options ♦ Enter numbers, 0-9, using single keystroke ♦ Enter alphanumeric data, A-Z, 0-9, SPACE, using 2 keystrokes per character

Probe Connections

The sample probe assembly is a hand-held device that enables you to take vapor samples at precise locations. It connects to the instrument by means of an umbilical. The umbilical has two quick-disconnect fasteners (one electrical, one sample line) at the instrument end. Use the slide-on connector, located at the forward end of the probe, to attach various sampling devices. The operator keypad and measurement display are also located on the handle, as shown in the following diagram.

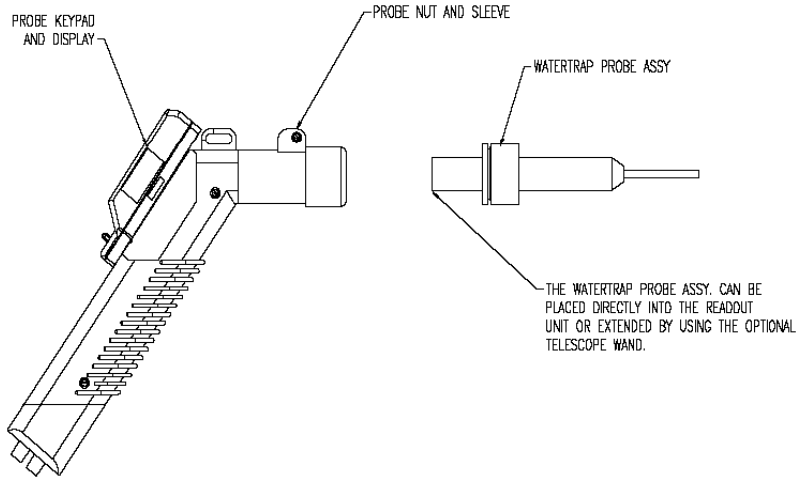


Figure 7. Sample Probe Assembly

BASIC Probe Display

The BASIC sample probe has a 4-character LCD display, as shown below, that displays measurement information. The display also contains an overrange indication, expressed as ">" when active. Three measurement unit types — ppm, ppb, or % — (selected during setup) are displayed to the right of the measurement data. Only those units selected during setup, however, are visible during survey.

INSTRUCTIONS CONTINUED ON NEXT PAGE

A segmented analog bargraph that represents a logarithmic scale for the total analyzer range appears below the digital display. This display, which may be backlit under low light conditions, is active only in the RUN mode. In all other modes, it displays *OFF*.

The measured value display area is used to flash logging prompts during survey and log mode. In addition, errors on the sidepack are indicated by "Err" on the probe. Errors are cleared by using the LOG key.

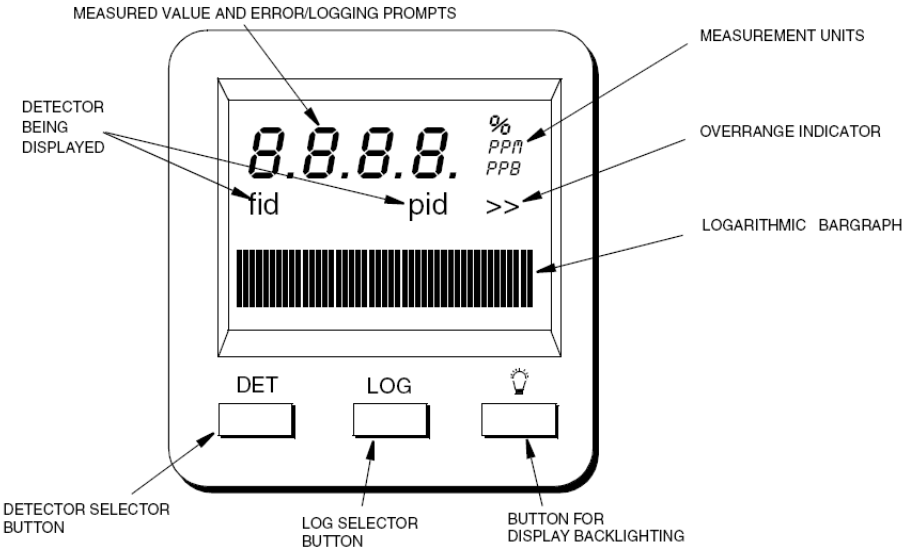


Figure 8. TVA-1000B BASIC Probe Display

NOTE: Use of the backlight draws additional power from the battery and will shorten the runtime of the TVA-1000B.

BASIC Probe Keys

The BASIC probe keypad has three keys. The keys are labeled with DET, LOG, and a lamp symbol.

Key	Function
DET	Press and release this key to select the display of either the FID or the PID readings in the RUN mode. Press and hold this key (approx. 2 seconds) to cancel selected logging modes.
LOG	Press and release this key to start the datalogging feature. Survey information is then automatically stored in the analyzer memory. The selection in SETUP/Log must be AUTO or VOC. Press and release this key to clear a sidepack error when the display reads " Err".
Lamp Symbol	Press this key to turn the backlight for the probe display on or off. You should use it only during low light conditions as it draws additional power from the battery and thus shortens instrument run time.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Quick Start Procedure

Before starting the unit, perform the following steps:

1. Charge battery.
2. Connect sample probe.
3. Fill/install hydrogen tank (FID versions).
4. Open the hydrogen valve (FID versions).

To start the unit, execute the following procedure:

1. Press ON.
2. Press CONTROL.
3. Press 3 to ignite.
4. Press 2 = Setup.
5. Press 1 = Calibrate
6. Press 2 = Span Concentration.
7. Enter Span Concentration for calibration gas being used and press EN accept.

NOTE: If PID only, enter concentration of isobutylene. If FID only, ent concentration of methane. If dual, enter concentration of both gases.

---

8. Press 3 = Zero.
9. Press 1 = Both.
10. Challenge analyzer with zero gas sample.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

11. Press ENTER = start.
12. Wait to stabilize.
13. Press ENTER to accept.
14. Press 4 = Span.
15. (PID 1st) Press 2 = PID.
16. Press ENTER = start.
17. Challenge analyzer with isobutylene span gas and wait for readings to stabilize.
18. Press ENTER to accept.
19. Press 4 = Span.
20. Press 3 = FID.
21. Press ENTER = Start.
22. Challenge analyzer with methane span gas and wait for readings to stabilize.
23. Press ENTER = Accept.
24. Press 5 = Response Factor.
25. Confirm that Response Factor says "RF0:DEFAULT"
26. Press EXIT 2 times to main menu.
27. Press 1 = Run.

You are now in the survey mode.

NOTE: To perform more sophisticated operations, you will need to read the rest of the manual .

To power down this instrument, simply press and hold the OFF key. With FID versions, you must also shut off the gas valve to avoid depleting the tank supply.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Calibration

The use of multipoint calibration and multiple response factors/curves with the TVA-1000B must be fully understood before employing these features. To help explain these TVA-1000B capabilities, three scenarios follow:

Scenario 1

To maximize standard accuracy, it is highly recommended that you calibrate with methane for the flame ionization detector and isobutylene for the photoionization detector. Almost all published response factors for FIDs and PIDs are based upon methane and isobutylene, respectively. By employing a *multipoint calibration* for these compounds, you will improve the accuracy of each detector over the *entire dynamic range*. Response factors/curves can then be employed for correcting the detector's response to different compounds. However, once a *multipoint calibration* has been employed, any *response curve* must characterize only the relative response at each concentration, excluding curvature of the calibrated compound. Thus , use of both multipoint calibration and response curves at the same time is difficult, and is not recommended.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Scenario 2

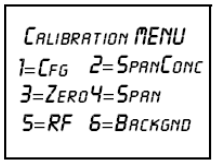
If, for example, you want to measure several different compounds over wide concentration ranges, it is best to use a *single-point calibration* and then enter *response curves* for each specific compound (up to 9 response factors/curves can be entered into the analyzer).

Scenario 3

If, instead, you want to measure in direct readings (response factor = 1) for one specific compound with maximum accuracy over a wide range of concentrations, perform a *multipoint calibration* with the specific compound. Up to 9 span points (plus zero) can be entered for each detector. The use of a response curve is thus unnecessary as the detector is already reading the direct PPM for that specific compound.

CAUTION: If you use multipoint calibration or a gas other than methane or isobutylene and then apply response factors/curves (that have been generated with reference to a single point methane/isobutylene calibration), the resulting measurements will probably be incorrect.

To provide the specified accuracy, the instrument must be calibrated at the beginning of each workday. To reach the CALIBRATION menu from the MAIN MENU, choose 2 = Setup and 1 = Calib. When you reach the CALIBRATION menu, you will see the following selections:



The steps involved in calibrating the TVA-1000B are as follows:

1. Configure the calibration variables *(Cfg)*.
2. Define the span concentrations to be used *(SpanConc)*.
3. Zero the instrument using either a zero gas or clean ambient air *(Zero)*.
4. Calibrate the reference point(s) using known span gases. The TVA-1000B can be configured for as many as nine (9) different span gas values *(Span)*.
5. **Optional**: Set instrument response factors if necessary *(RF)*.
6. **Optional**: Take background reading *(Backgnd)*.

NOTE: Prior to performing calibration, the instrument must be on and warmed up for approximately 30 minutes. The pump must be ON, the PID lamp must be ON, and the FID must be ignited throughout the warm-up period.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Detector Counts

Detector counts are the raw, unscaled detector output values associated with a gas measurement performed by the FID or the PID. Before a detector reading is displayed or recorded, the detector signal is converted from analog to digital. The result is a raw number, or A/D counts.

When a detector is calibrated, the detector counts for the zero gas and each of the span gases are saved in memory. These detector counts are then used as reference points for calculating the concentration values to be displayed or stored.

When calibrating the TVA-1000B in the "Manual" accept mode, the counts from the last calibration (Zero or Span) are displayed before the calibration process is initiated. Once the calibration process is initiated, the live detector counts are then displayed. You can refer to these counts as an indication of when the reading has stabilized, or as a means of tracking the repeatability of your calibrations.

You can also use these counts as an indication of the success of a calibration. The "zero" counts are the counts expected when a zero gas is applied to the detector. The span counts are the counts expected when a span gas of known concentration is applied to the detector. Finally, the detector sensitivity can be calculated by subtracting the zero counts from the span counts and dividing by the span gas concentration. Use the following general observations as a guideline:

Detector	Zero Counts	Detector Sensitivity
FID	<5000	160-260 counts/ppm Methane
PID (10.6 eV lamp)	2000-8000	3500-6000 counts/ppm Isobutylene
PID (11.8 eV lamp)	2000-20,000	300-900 counts/ppm Isobutylene

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Example: A TVA-1000B FID is calibrated with zero air and a 100 ppm Methane in air span gas. The counts observed for the zero are 2895 and the counts observed for the span are 27395. The span sensitivity is thus 245 counts/ppm [(27395-2750)/100 ppm]. Since both of these values (2895 zero counts and 245 detector sensitivity) are within the acceptable range, the calibration is a good calibration. Examples of a bad calibration include unusually high zero counts, or unusually low detector sensitivity. These problems can often be attributed to poor calibration gases, contaminated sampling accessories, a faulty detector capsule, or failure to follow the proper calibration procedure. For more information, consult the "Troubleshooting" guide in this manual or contact TEI for assistance.

Defining the Span Gas Concentration(s)

NOTES:

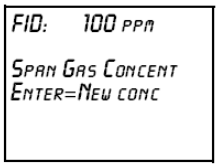
1. The span gas concentration is the known concentration of the gas standards used to calibrate your TVA. Methane in air is the recommended calibration standard for the FID, and Isobutylene in air is the recommended calibration standard for the PID. Other gases may be used if desired.

2. If your instrument is equipped with dual detectors, you may choose to calibrate the PID and FID separately or together.

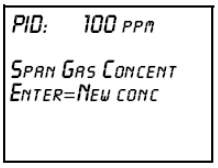
3. If your instrument is configured for multiple span points, be sure to set the concentration for ALL span points.

1. From the CALIBRATION menu display, press 2=SpanConc. The upper display (or two displays if the unit is a dual detector version) will display the concentration value of your span gas (expressed as ppb, ppm, or %) as of the last calibration:

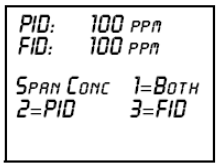
FID only



PID only

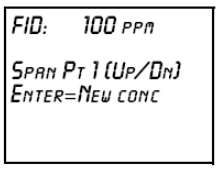


FID/PID

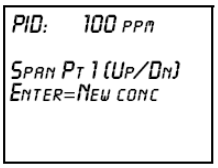


If the TVA-1000B is configured for multiple calibration points, the span gas concentration values for Point #1 will be displayed. The Up and Down arrow keys can be used to scroll through the span gas concentration values for other points:

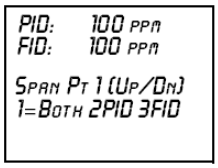
FID only



PID only



FID/PID



INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Troubleshooting

Problem	Possible Reason	Solution/Action
Unit will not turn on	Battery charge low	Charge battery.
	Bad battery connection	Ensure proper battery connection.
	Bad battery	Replace battery.
	Blown fuse or faulty keypad	Contact TEI authorized service center.
Pump won't turn on	Defective pump Faulty keypad	Contact TEI authorized service center.
Low pump flow	Clogged sample intake	Clean/replace filter cups. Clean/replace sample line. Clean/replace flame arrestor.
	Pump fault	Contact TEI authorized service center.
Keypad will not respond	Faulty keypad	Contact TEI authorized service center.
Probe display blank or probe buttons will not respond	Faulty probe/display assembly	Replace probe/display assembly. Contact TEI authorized service center.
FID won't ignite	Insufficient sample flow	Turn pump on. Clean/replace filter cups. Clean/replace flame arrestor.
	Hydrogen valve off	Turn on hydrogen supply valve and allow 1-2 minutes before igniting.
	Insufficient hydrogen supply (<500 psi)	Check hydrogen gauge on supply tank. Refill tank if necessary.
	Hydrogen leak	Check low pressure hydrogen output gauge. If <10.5 psi, contact TEI authorized service center.
	Broken igniter	Inspect igniter coil on FID capsule for breakage. If broken, replace capsule.
	Dirty igniter contacts	Inspect/clean igniter contacts (NOT igniter coils).
	Battery charge low	Charge battery pack.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Problem	Possible Reason	Solution/Action
FID noisy	Water/contamination in the detector chamber	Clean/replace FID capsule and flame arrestor.
		Clean/replace filter cups.
	Erratic pump flow	Clean/replace flame arrestor.
		Ensure proper calibration.
	Bad calibration	
Unable to calibrate FID	FID flame out	Ignite FID.
	Span concentration not properly set	Input correct span gas concentration at CAL menu.
	Cal gases contaminated	Use clean cal gases and sampling equipment.
	Sample line/filter cups contaminated	Clean/replace sample line. Clean/replace filter cups.
	FID capsule contaminated or faulty	Clean/replace FID capsule.
	Contaminated hydrogen tank	Replace hydrogen tank.
	Internal detector fault or contamination	Contact TEI authorized service center.
Excessive hydrogen consumption (<8 hours of run time for 2200 psi hydrogen)	Insufficient hydrogen pressure	Refill tank.
	Leaking hydrogen tank	Replace tank.
	Internal hydrogen leak	Contact TEI authorized service center.
Flameout problems	Sample hydrocarbon content too high	Use dilutor kit to achieve concentration within the dynamic range of the TVA.
	Insufficient oxygen in the sample (<14%)	Use dilutor kit to dilute sample with air containing sufficient oxygen. Use PID for measurements.
	FID capsule contamination	Clean/replace FID capsule.
	Insufficient sample flow	Clean/replace filter cups. Clean/replace flame arrestor.
Moisture at FID flame arrestor *NOTE: Normal operation produces some moisture. If performance is affected, attempt these solutions.*	Insufficient sample flow	Clean/replace filter cups. Clean/replace flame arrestor.
	Insufficient warmup time	Allow 15-20 minutes warmup.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Problem	Possible Reason	Solution/Action
PID lamp not operating	Bad PID lamp	Replace PID capsule.
PID noisy	Bad PID lamp	Replace PID capsule.
	Dirty PID window	Clean PID window.
	Erratic pump flow	Clean/replace filter cups. Clean/replace flame arrestor.
	Bad calibration	Ensure proper calibration.
Unable to calibrate PID	Pump not on	Turn pump on.
	PID lamp not on	Turn PID lamp on.
	Span concentration not properly set	Input correct span gas concentration at CAL menu.
	Cal gases contaminated	Use clean cal gases and sampling equipment.
	Sample line/filter cups contaminated	Clean/replace sample line. Clean/replace filter cups.
	PID window dirty or capsule contaminated or faulty	Clean PID window. Replace PID capsule.
	Internal detector fault or contamination	Contact TEI authorized service center.
Slow response time	Insufficient sample flow	Clean/replace filter cups. Clean/replace flame arrestor. Clean/replace sample line.
	Sampling non-volatile compounds	Contact TEI Applications Laboratory.
High background readings	High ambient concentration	N/A.
	Zero drift/improper calibration	Ensure proper zero/span calibration.
	Sample line contamination	Clean/replace sample line. Clean/replace filter cups.
	Detector capsule contamination	Clean/replace FID capsule. Clean PID window. Replace PID capsule.

6.0 Calibration and Use of the RKI Eagle Combustible Gas Indicator - Tool Watch # 205013-016

6.1 Introduction

The RKI Eagle is the most advanced portable gas detection system available. The Eagle is built for rugged reliability and ease of use and includes the latest innovations in gas detection technology:

• Simultaneous detection of one to six gases. Standard target gases include combustible gas (% LEL and ppm), oxygen deficiency, carbon monoxide, and hydrogen sulfide.

• Powerful sample-drawing pump with up to 100-foot range.

• Dot-matrix liquid crystal display (LCD) for complete, understandable information at a glance.

• Microprocessor control for all functions, including data logging and user-adjustable alarms.

• Visible and audible alarms for hazardous conditions and malfunctions.

• UL and CSA classified. Intrinsic safety for Class I, Division I, Groups A, B, C, and D hazardous atmospheres (standard 4-gas model and non-standard toxic gas versions). Consult RKI Instrument, Inc., for classification of other Eagle versions.

• Tough case with a balanced, light-weight design.

WARNING: The Eagle detects a combination of combustible gas, oxygen deficiency, hydrogen sulfide and carbon monoxide, or other toxic gases which can be lethal. Users must follow the instructions and warnings in this manual to assure proper and safe operation of the Eagle.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Table 2: Standard Sensor Specifications

	Combustible Gas (%LEL[1])	Combustible Gas (PPM[2])	Oxygen	Hydrogen Sulfide	Carbon Monoxide
Range	0 to 100% LEL	Depends on target gas[4]	0 to 40% O_2	0 to 100 ppm	0 to 500 ppm
Alarm 1	10% LEL	5000 ppm	19.5% O_2 (decreasing)	10.0 ppm	25 ppm
Alarm 2	50% LEL	25,000 ppm	23.5% O_2 (increasing)	30.0 ppm	50 ppm
TWA Alarm	N/A	N/A	N/A	10.0 ppm	25 ppm
STEL Alarm	N/A	N/A	N/A	15.0 ppm	400 ppm
Detection Principle	Catalytic combustion	Catalytic combustion	Electro-chemical	Electro-chemical	Electro-chemical
Response Time (to 90%)[5]	30 seconds	30 seconds	30 seconds	30 seconds	30 seconds
Accuracy (of fullscale)	± 5%	± 5%	± 5%	± 5%	± 5%

1 LEL (Lower Explosive Limit)
2 PPM (Parts Per Million)
3 Alarms settings are user adjustable. See "Updating the Alarm Point Settings" on page 32.
4 The PPM range represents the same range as 0 to 100% LEL for that gas. For example, 100% LEL for methane = 5% by volume = 50,000 PPM. Therefore, the PPM range for methane is 0 to 50,000.
5 With the Eagle's standard hose and probe attached.

INSTRUCTIONS CONTINUED ON NEXT PAGE

6.2 Sensors and Switches

This section describes the Eagle's standard sensors and switches. Non-standard sensors are described in Appendices C, D, and E. Your specific Eagle model may not include all of the sensors described below. Under normal conditions, the Eagle's standard sensors have an operating life of approximately two years.

Combustible gas sensor
The combustible gas (LEL) sensor is mounted with the flame arrestor down in the sensor block to allow the sample flow to diffuse into the sensor. Five pins extend from the top of the sensor. The sensor cable connects to the pins on one end and terminates in a four-position connector, which plugs into the **COMB** socket on the analog print circuit board (PCB). The LEL sensor detects combustible gas and vapors in the atmosphere with a catalytic platinum element. The reaction of gas with oxygen on the catalyst causes a change in the resistance of the element, which is converted by the Eagle into a reading of combustible gas concentration.

Oxygen sensor
The oxygen (O2) sensor is mounted face down in the sensor block to allow the sample flow to diffuse into the sensor. A multi-pin plug connects the O2 sensor to the **CN2** socket on the analog PCB. The O2 sensor is an electrochemical cell, which reacts to the oxygen in the atmosphere and produces a voltage proportional to the oxygen concentration. This voltage is converted by the Eagle into a reading of oxygen concentration.

Standard toxics (CO and H2S) sensors
The CO and H2S sensors are physically very similar. They have cylindrical bodies and are mounted face down in the sensor block. A three-position connector from each sensor plugs into **EC1** or **EC2** socket on the analog PCB. The sensor connected to the **EC1** socket displays as channel 3; the sensor connected to the **EC2** socket displays as channel 4.

The toxics sensors are electrochemical cells, which react to the target gas in the atmosphere, producing a current proportional to the concentration of gas. The current is converted by the Eagle into a reading of target gas concentration.

Methane Elimination Switch
The methane elimination switch (SW1) is mounted near the top right corner of the main PCB.

For applications where methane is an interfering gas, you can set the methane elimination switch to eliminate most response to methane (see "Appendix B: Methane Elimination" on page 52). An *external* methane elimination switch is available as an *option*.

CAL/SETUP Switch
The CAL/SETUP switch (SW2) is mounted near the middle left edge of the main PCB. This switch controls the Eagle functions available to the user by disabling the SHIFT/t button. Without the use of this button, the user is unable to enter Calibration or Setup mode. (Display mode is available with either switch setting.) See "Setting User Access" on page 13 to change the switch setting.

6.3 Operation

The Eagle has four operating modes: normal operating mode, display mode, setup mode, and calibration mode. This section describes the Eagle in normal operating mode. It includes procedures to start up the Eagle, set various detection options for the combustible gas channel, and shut down the Eagle.

NOTE: The screens illustrated in this section are intended as examples only. The screens displayed by your Eagle model may be slightly different.

Starting Up the Eagle

1. Connect the sample hose to the Eagle's quick connect inlet fitting.

2. Connect the hydrophobic filter and probe tip to the sample hose's quick connect fitting.

3. Press and briefly hold down the POWER/ENTER button. If the Lunch Break feature is on (see page 33), the Resume Datalog screen displays. (If the Lunch Break feature is off, the Battery Voltage screen displays.)

- Press the AIR/s button to continue accumulating time weighted average (TWA) and PEAK readings from the last time the Eagle was used. (The short-term exposure limit [STEL] reading is reset each time the Eagle is turned on). The Battery Voltage screen displays.
- Press the DISP/ADJ button to restart these measurements. The Battery Voltage screen displays if you do not press the AIR/s or DISP/ADJ button within 5 seconds, the Eagle automatically resumes datalog readings and displays the Battery Voltage screen.

The Battery Voltage screen displays the minimum usable and actual battery voltage (for example, 6.0V). If the battery voltage is too low, the Eagle will not continue.

NOTE: The following screen only displays if the data logging option is installed. If the data logging option is not installed, the Self Diagnosis screen displays after the Battery Voltage screen. This message displays the date and time as set in Setup mode. The data logging option uses this information to record the time and date of sample and alarm events. The following two screens display while the Eagle checks itself for proper operation. The Eagle alerts you if a malfunction occurs. When the Eagle successfully completes its self check, the **OK** message displays in place of the **STAND BY** message, then the normal operating screen displays. The normal screen displays fresh-air concentrations for all gases. The Eagle sounds a double tone to indicate it is in normal operation.

CAUTION: *Do not use gas from a cigarette lighter to test response to combustibles. Exposing the combustible gas sensor to uncontrolled high concentrations of gas will reduce response and sensor life.*

The Eagle continuously monitors the sampled atmosphere and displays the gas concentrations present for its target gases. In a lowlight environment, press any button to turn on the display backlight. (See "Updating the Back Light Setting" on page 36 to program backlight duration). If

the Confirmation Beep option is turned on, the Eagle beeps once every 15 minutes to verify that it's on the job. To use the probe, insert it into the monitoring area and wait a few seconds for response.

NOTE: Response time increases with the length of the sample hose. Very long sample hoses may require several seconds to show response at the Eagle.

Monitoring Combustible Gas in the PPM Range

1. Start the Eagle in the LEL range as described in "Starting Up the Eagle" on page 9.
2. Allow the combustibles sensor to stabilize (3 to 5 minutes). This stabilization period is required for the PPM range only.
3. Press the LEL/PPM button. The Eagle displays **PPM** in place of **LEL%** for combustible gas, and the gas reading displays in parts per million. 4. If the PPM reading is not zero, take the Eagle to a fresh air environment, then perform the demand zero procedure as described in "Preparing for Calibration" on page 41.

NOTE: For the data logging **option**, combustible gas readings are logged in %LEL regardless of the LEL/PPM setting.

Monitoring Combustible Gases Other than Methane

If the combustible gas sensor is calibrated to methane (CH4), use Table 4 to determine the concentration of combustible gases other than methane. This table is based on Eagles in full response mode (methane elimination switch set to **CH4**) and calibrated to methane. Multiply the display reading by the factor in the appropriate column in the table. For example, if you are using the Eagle to detect hexane and the display reads 10% LEL, the actual hexane reading is 10% x 3.00 = 30% LEL hexane.

WARNING: The Eagle's alarms are initiated by the DISPLAY reading not the FACTORED reading. If you are monitoring for hexane as in the example below and the low alarm is set for 10% LEL, the Eagle will initiate a low alarm at 30%LEL hexane (display reading of 10% LEL).

To determine the concentration of other combustible gases with the Eagle in methane elimination mode, see Table 4 on the next page.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Table 4: Full Response Mode Conversion Factors (Methane Calibration)		
Target Gas	**LEL Factor (Methane Calibration)**	**PPM Factor (Methane Calibration)**
Benzene	2.80	0.67
Ethane	1.40	0.84
Ethanol	2.25	1.5
Ethylene	1.58	0.4
Hexane	3.00	0.67
Hydrogen	1.65	1.4
IPA	2.83	1.13
Isobutane	1.93	1.21
MEK	3.00	1.08
Methane	1.00	1.00
Methanol	2.33	2.57
Propane	2.30	0.92
Propylene	2.00	0.80
Toluene	2.80	0.67

INSTRUCTIONS CONTINUED ON NEXT PAGE

6.4 Calibration

Calibrate the Eagle when you replace a sensor. Also calibrate the Eagle periodically to assure proper sensor response. You can program the Eagle to notify you when it is due for calibration (see “Updating the Time Calibration Setting” on page 38). The frequency of calibration depends upon the amount and type of use. A typical calibration frequency is once per month.

Calibration Supplies and Equipment
To calibrate the Eagle, you need:
• Known calibrating samples of combustible and the appropriate toxic gases. The samples should have concentrations in approximately the middle of the range of detection.
• An oxygen-free source, such as 100% nitrogen or CO in a nitrogen balance
• A demand-flow regulator to provide adequate sample gas flow for one-source, auto-calibration, you can use the RKI Four-Gas Cylinder to adjust all the sensors at the same time, automatically, with no need for a zero-oxygen source. This section includes instructions for calibration with the demand-flow regulator and RKI Four-Gas Cylinder. This section also includes instructions for calibration with individual gas sources.

Preparing for Calibration
1. Take the Eagle to a non-hazardous location with fresh-air conditions.
2. Turn on the Eagle and allow one minute for warm up.
3. Press and hold the AIR/s button until a tone sounds.
The Eagle automatically sets the combustible gas and toxics circuits to zero and the oxygen circuit to 20.9%.
4. Screw the regulator to the calibration cylinder.
5. Connect the calibration tubing to the regulator.

Calibrating the Eagle
Press and hold the SHIFT/t button, then press the DISP/ADJ button. The Calibration menu displays.
NOTE: The following screens illustrate a four-gas Eagle with the data logging option and are intended as examples only. Your Eagle may display slightly different screens. The Eagle’s Calibration menu includes two methods of calibration:

Auto Calibration and Single Calibration
• **Auto Calibration:** This method allows you to calibrate all four sensors simultaneously. It is designed for use with the RKI Four-Gas Calibration Cylinder and is the quickest and easiest method to calibrate the Eagle.
• **Single Calibration:** This method allows you to calibrate one sensor at a time. Use this method if you are only calibrating one or two sensors, if you are calibrating non-standard toxic sensors, or if you are not using the RKI Four-Gas Calibration Cylinder.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Calibrating with the Auto Calibration method

This section describes calibration using the Auto Calibration method. To calibrate using the Single Calibration method, see "Calibrating with the Single Calibration method" on page 44.

1. Use the AIR/s and SHIFT/t button to place the prompt next to the **AUTO CALIBRATION** menu option.
2. Press the POWER/ENTER button to display the Calibration Values screen. The gas concentrations displayed in the Calibration Values screen must match the gas concentrations listed on the Four-Gas Calibration Cylinder. If *all* concentrations match, go to step 7. If *one or more* concentrations do not match, continue with step 3.
3. To adjust the values on the screen, hold down the SHIFT/t button, and press the DISP/ADJ button. The Auto Calibration screen for the combustible gas channel displays.
4. Use the AIR/s (increase) and SHIFT/t (decrease) buttons to set the correct combustible gas value.
5. Press the POWER/ENTER button to enter the new setting. The Auto Calibration screen for the next channel displays.
6. Repeat steps 4 and 5 to set the correct values for the remaining channels and return to the Calibration Values screen.

NOTE: The RKI Four Gas Cylinder contains approximately 12% O2 by volume. Be sure to set the "OXY" reading to agree with the concentration listed on the cylinder's label, not zero.

7. With the Calibration Values screen displayed, press the POWER/ENTER button. The gas readings flash.
8. Connect the tubing from the regulator to the Eagle's probe. Wait approximately 1 minute or until the readings stabilize.
9. Press the POWER/ENTER button to set the calibration to the programmed values.

If a sensor(s) cannot calibrate to the proper value, **FAIL PUSH AIR KEY** displays and the Eagle lists the sensor(s) that failed to calibrate. (The other sensors calibrate normally.) The buzzer and alarm lights activate. Press the AIR/t button to reset the alarm and return to the Calibration menu. Replace the failed sensor(s), then repeat calibration.

10. **AUTO CALIBRATION END** displays, then the Calibration menu displays.
11. Disconnect the tubing from the probe.
12. Unscrew the regulator from the calibration cylinder.
13. Press the SHIFT/t button to place the prompt next to the

NORMAL OPERATION menu option, then press the POWER/ENTER button to return to the normal screen.

INSTRUCTIONS CONTINUED ON NEXT PAGE

Calibrating with the Single Calibration method

This section describes calibration using the Single Calibration method. To calibrate using the Auto Calibration method, see "Calibrating with the Auto Calibration method" on page 42.

CAUTION:*The single calibration method does not have a "FAIL" notification. Replace sensors that cannot be set to agree with the calibration source, then recalibrate.*

1. Use the AIR/s and SHIFT/t buttons to place the prompt next to the **SINGLE CALIBRATION** menu option.
2. Press the POWER/ENTER button to display the Single Calibration menu.
3. Use the AIR/s or SHIFT/t button to place the prompt next to the channel to calibrate (in this example the combustible gas channel).
4. Press the POWER/ENTER button. The Single Calibration screen displays for the channel you selected. The gas reading flashes.
5. Connect the tubing from the regulator to the Eagle's probe.

NOTE: The combustible gas sensor is a general hydrocarbon sensor that responds to most flammable vapors and gases; the response will vary depending upon the substance. For best results, calibrate the Eagle to the target gas or vapor
.

6. If necessary, use the AIR/s (increase) and SHIFT/t (decrease) buttons to adjust the reading to match the concentration listed on the calibration cylinder.
7. Press the POWER/ENTER button to set the span value. **SINGLE CALIBRATION END** displays, then the Single Calibration menu displays.
8. Disconnect the tubing from the probe.
9. Repeat steps 3 through 8 for any other channels you want to calibrate. Make sure you use an appropriate calibration cylinder for each channel.

CAUTION:*When calibrating the oxygen channel, verify the concentration of oxygen listed on the cylinder's label. For oxygen-free samples (100% nitrogen for example), set the oxygen span setting to 0.0%.*

10. After the last channel is calibrated, disconnect the calibration tubing from the probe, then unscrew the regulator from the calibration cylinder.
11. With the Single Calibration menu displayed, press the SHIFT/t button until the prompt is next to the last channel, then press the SHIFT/t button again. The **ESCAPE** message displays.
12. Press the POWER/ENTER button to return to the Calibration menu.
13. Press the SHIFT/t button to place the prompt next to the **NORMAL OPERATION** menu option, then press the POWER/ENTER button to return to the normal screen.

7.0 Calibration and Use of the Gastech GT 201 Oxygen and Combustible Gas Meter - Tool Watch # 205010

7.1 Introduction

The Gastech 200 series instrument has been designed to detect toxic and flammable vapors and give the operator a warning before they reach harmful conditions. **In order to ensure that the instrument will warn the user of dangerous concentrations, it is essential that the instructions be understood and followed.** The electrochemical oxygen detector in the instrument detects oxygen concentrations in the 0-30% range. The instrument also detects hydrocarbons in the 0-10,000 ppm range. Combustible gases are detected in 0-100% of their lower explosive levels (LELs) and are catalytic compensated. The response time for detection is approximately 5 seconds with the five foot hose attached. The Gastech 201 is intrinsically safe and has a Class I, Division 1, Groups A, B, C, and D Safety rating The preferred temperature range for operation is – 4 ^{o}F to 113 ^{o} F. The instrument can be operated in relative humidity ranges of 0-95%.

7.2 Control Panel

The operator control panel is on the top of the monitor. The panel consists of six embossed control buttons. The functions of each button are described below:

ON/OFF-is a push button which turns the monitor on or off when pressed. Press the button to turn on the monitor. Press and hold for at least five seconds to turn the monitor off.

RESET-serves many purposes. It is used to reset latching alarms, and is also used to restart the pump if it shuts down due to a low flow condition, to exit, or go to previous screens in function menus.

FUNC/+ - is used to scroll through various options or set-up parameters. The "+" function of the button, when active is used to increase the setting during calibration, and is also used as "YES" button during other operations.

BACK LITE- pressed during normal operations to illuminate the display for up to 60 seconds. The "minus or – " function of the button is used to decrease the setting during calibration, and is also used as a "NO" button during other operations.

ADJUST/ENTER- used to adjust the monitor to "fresh air" readings. The ENTER function of the button is used to accept calibrations or other parameters, when the GT 201 is in the mode for these types of adjustments.

LEL/PPM-is used for switching between LEL and hydrocarbon ppm readings on models that have the catalytic (LEL) sensor.

7.3 Operation

The most common modes of operation for use at Bay West projects will be discussed in this section. For complete explanation of the modes refer to the manufacturer's operator manual.

7.3.1 Start up

- Install four "D" size batteries. If alkaline batteries are used make sure the slide switch at the bottom of the battery compartment is set to the ALK position. If re-chargeable batteries (Ni-Cad) are used the switch must be set to NI-CAD. To achieve correct polarity, install the batteries according to the diagram engraved on the bottom of the battery compartment.
- Ensure that the hydrophobic filter is in good condition, and installed in the probe body.
- Attach the probe to the female disconnect coupler fitting on the sample hose. Attach the other end of the hose to the female disconnect coupler fitting on the front of the instrument.

Fresh Air Calibration

- Press the ON/OFF button once, then release. The display temporarily shows the software version of the monitor and the number of data logging hours that remain in memory. During the warm-up period, installed sensors stabilize. The pump can be heard and the display indicates: WARMING UP. The red LED blinks slowly during this process. Allow one minute for warm-up. The LED will stop blinking once the unit is stable.
- The unit will respond with a periodic beep and display : WARM UP COMPLETE
- Perform the demand zero by observing the display, press and hold down the ADJUST/ENTER to adjust the meter to "fresh air" readings. Once the display reads: DONE THANK YOU release the ADJUST/ENTER button.
- Verify that the display reads 000 in the %LEL/PPM field and 20.9% in the % OXY field.
- Confirm the normal operation of the oxygen detection by breathing through your mouth into the sample probe. The displayed reading should decrease until it reaches 19.5 % oxygen, activating the oxygen alarm in the process. The alarm will shut off and the % OXY reading will blink until the concentration reaches 20.9%

Span Gas Calibration

- Connect the 5-foot hose to the inlet fitting and connect the probe to the end of the hose. Turn on the GT201 by pressing the ON/OFF button. Allow at least a minute for the instrument to stabilize. The unit is stabilized once the red LED quits blinking, and the display reads WARMUP COMPLETE.
- Press ADJUST/ENTER once. The display will go to the main screen.
- To enter calibration mode press the RESET and BACK LITE/- buttons together three times. The display shows the initial calibration mode field:

Version N.NN
Calibrate

- Press ADJUST/ENTER to begin the calibration procedure. The first field to come up is for zeroing the PPM COMB channel. The GT201 displays:

Zero Gas
NNNN PPM COMB

- Either use the FUNC/+ or the BACKLITE/- to bring the display reading to 0000 PPM COMB.
- Once zeroing is complete, press the ADJUST/ENTER button to save the zero setting. The GT201 now displays:

Zero Gas

N.NN % VOL OXY

- Install a Tedlar bag filled with nitrogen gas (0.00% oxygen) in line with the sampling probe.
- Allow at least one minute for the reading to stabilize then use either the FUNC/+ or the BACK LITE/- buttons to bring the display reading to 0.00% VOL OXY.
- Press ADJUST/ENTER to save the zero setting. The GT201 will now display:

Span Gas
NNN % LEL COMB

- Install a Tedlar bag filled with 2.5 % methane (natural gas) in line with the sampling probe.
- Allow enough time for the instrument to stabilize. Press FUNC/+ or BACKLITE/- to bring the display to read 50% LEL COMB (the value indicated on the calibration gas cylinder).
- Press ADJUST/ENTER to save the span setting.
- Disconnect the span gas bag and allow the instrument to purge fresh air for at least 90 seconds.
- Using either FUNC/- or BACK LITE/- bring the display reading to 20.9% VOL OXY for the oxygen sensor.
- Press ADJUST/ENTER to save the span setting. The calibration process is now complete
- The GT201 displays:

Exit
Press Any Key….

- Press any button to exit calibration mode.

The GT201 is now ready for normal operation. The unit periodically sounds a short beeping tone. This comfort beep is simply an indication that the monitor is working properly. During normal operation the unit will simultaneously detect both combustible gases and oxygen content.

7.4 Documentation

All calibration results, repairs and maintenance must be documented in the instrument's log book which is inside the carrying case. This provision is required for some government contracts and is a good industry practice. If the log book is missing please contact one of the health and safety specialists for a replacement.

8.0 Calibration and Use of the Thermo Environmental Instruments - Model 580B PID Organic Vapor Monitor - Tool Watch # 200021 and 200018

8.1 Introduction

The 580B is a portable organic vapor meter which detects and quantifies most organic vapors with a highly sensitive photoionization detector (PID). The operating range is 0-200 parts per million (ppm) with a minimal detectable level of 0.1 ppm. There are three lamps available for this instrument 10.0eV, 10.6 eV and 11.8 eV. Knowing the ionization potential of the compound of interest will enable the user to select the correct lamp. However, it will not give any information as to the performance of the detector in its ability to measure a specific compound. The following is an idealized response chart to be used as a qualitative guideline.

Decreasing PID Response:	Aromatic Compounds
	Unsaturated Compounds
	Saturated Compounds
	Ketones
	Alcohols
	Compounds with Subgroups

The true test of the instrument performance is performed by measuring the specific compound of interest and compare its response to a good performing standard such as isobutylene. The relative comparison with isobutylene is an effective way of measuring a variety of compounds without the need to recalibrate for each individual compound. All calibration and subsequent response factor developments should be done with the same lamp/instrument configuration. Once a response factor is generated (e.g. benzene) , it is entered into the 580 OVM.

8.2 Program Operation

The **580** has several switches located just below the display. They are labeled:

ON/OFF MODE/STORE RESET LIGHT+/INC -/CRSR SPKR

The **ON/OFF** switch toggles the lamp and pump power between on and off.

The **MODE/STORE, RESET, +/INC,-/CRSR, and SPKR** switches all have various meanings depending upon the instrument mode

Normally, the **SPKR** switch is used to toggle the instrument speaker (which behaves similar to a Geiger click) between the on and off positions.

Pressing **MODE/STORE** switch will cause the instrument to return to the Run mode, except when the instrument is already in this mode.

The **LIGHT** switch is used to illuminate the display.

The **580 OVM** has several modes. Some of these modes have sub-modes. The modes and sub-modes available with this instrument are summarized below:

MODE: Run Mode RUN SUB-MODES: Concentration meter normal, Max Hold
MODE: Log Mode
MODE: Parameter Mode
PARAMETER SUBMODE: Calibration Mode
MODE: Access Mode
MODE: Clock Mode
MODE: Communication Mode

The most common modes encountered for use at Bay West jobs will be explained in this text. For complete explanation of all of the modes refer to the manufacturer's instruction manual.

8.2.1 Instrument Start-Up

Power for Lamp and Pump

1. Turn on the instrument electronics by pressing ON/OFF switch.
2. Instrument display will indicate that the lamp is not lit. Pressing the ON/OFF switch again will tell the internal microprocessor to send power to the lamp and the sample pump. The system will do a self-check to insure that the lamp is lit. If not the instrument will retry. If after 14 seconds the lamp will not light, the display will indicate a lamp out condition. Check the seating of the lamp to see if it is tight. If the problem persists the unit will probably have to be sent to the manufacturer for service.
3. Once the lamp is lit, the display will show the ppm on the bottom line. The top line of the display will either be a bar graph or the maximum reading.
4. To turn the lamp and pump off, press the ON/OFF switch.

Run Modes

The instrument has 2 run modes, Max hold and Concentration meter. The run mode can be selected in the Parameters section. In the concentration meter mode, the top line will display a bar graph. The graph is a logarithmic bar graph over the range of 0-200 ppm. The bottom line will indicate numerically the current ppm.

In the Max hold mode the top line of the display will indicate the maximum reading. The bottom line will indicate the current ppm. Whenever a new maximum is detected the top line will be updated. The Max hold reading can be reset by pressing the RESET switch while in the reset mode.

Log Modes

The instrument has the ability to store readings for later analysis. Up to 700 readings can be stored. The logged data can be sent to a printer or compute with the cable accessories Typically, the projects encountered at Bay West do not involve such complex data tracking. For more details refer back to the manufacturer's instruction manual.

Speaker

While the instrument is in the Run mode the speaker may be turned on. The speaker generates a clicking response which will increase in speed as the concentration encountered increases (much like a Gieger counter). The speaker can be turned of or on by pressing the SPKR switch.

Low Battery

The instrument will display a warning sign when the battery voltage is low. The warning will be a flashing "B" in the left hand corner of the display. The unit should be re-charged when this occurs. To protect internal circuitry attach the charger line to the unit before plugging the charger into an electrical outlet. The unit can also be run with the charger in place.

Overrange

The instrument will display an overrange warning if the concentration goes above 2000 ppm. The top line of the display will show: OVERRANGE. Once this occurs the instrument will lock-out and this message will be displayed until the unit is moved to a clean area (organic vapors < 20 ppm).

Alarm

The instrument has an alarm which will sound if the ppm rises above a preset mark. The setting can be changed while in the parameters mode.

Main Menu

By pressing the MODE/STORE switch from the run mode and then pressing the -/CSR switch when asked if logging is desired, the instrument will display the main menu:

R/COM -/PARAM
+/ACCESS S/CLOCK

The other four operating modes can then be entered from the main menu. The user can return to operating mode by pressing the MODE/STORE switch.

Parameters Mode

All of the operation parameters can be entered or changed when the instrument is in the parameters mode. Care must be taken so that the user doesn't enter this mode by accident and unintentionally change the parameters for typical field use. The parameters mode can be entered by pressing the -/CRSR switch from the main menu. The are nine different sections in the parameters mode.

- Run Mode Selection
- Auto Logging Section
- Location Mode Selection
- Average Time Selection
- Alarm Setting
- Lamp Selection
- Response Factor Setting
- Calibration
- Free Space Indication

Pressing the +/INC switch will advance the instrument to the next section.

Pressing the -/CRSR will advance the instrument to the previous section

The +/INC and -/CRSR each have a different meaning depending upon what sub-section of the above sections you are currently in. The manufacturer's complete instruction manual provides more detail for each section and sub-section not included in this text.

Run Mode Selection-Selects Concentration meter normal and Max Hold. This is usually set at Concentration meter normal as a default for Bay West field surveys.

Auto Logging-Set to OFF as a default

Location Mode-Generally not used

Average Time Selection-Configured usually to update once every second
Time format M:SS (M in minutes, SS in seconds) AVERAGE = 0:01
Alarm Setting-Pre-established at the Office

Lamp Selection-Set to the current lamp installed (+/10eV). Do not change this setting without installing a different lamp!

Response factor setting-The current response factor setting will be displayed. The default is 1.00 for isobutylene. Benzene is the only other chemical we have established a different response factor for.

Calibration-Must be performed every day before use. Refer to the next section for details.

Free Space Indication-Gives a rough indication of the available memory for saving data log points.

Access Mode-For pre-setting security codes. Do not alter from of position.

8.2.2 Calibration

The instrument has been tested for calibration and linearity at the factory. However, the user must re-calibrate the instrument every time before use. 100 ppm isobutylene is the calibration gas chosen for this purpose. The calibration can be performed in the following steps:

1. Plug in the key chain into the RUN/CHG opening with the red line pointing up.
2. Turn on the electronics by depressing the ON/OFF button.
3. Light the lamp and turn on the pump by depressing ON/OFF again
4. Push MODE/STORE button

   Display will read LOG THIS VALUE

   MAX PPM = 000
5. Push - /CRSR

   Display will read: R/COMM -/PARM

   +/ACCESS S/CLOCK
6. Push - /CRSR

   Display will read: CONC METER

   MAX HOLD
7. Push - /CRSR

   FREE SPACE= 9792 (or some number close to that)
8. Push - /CRSR

   Display will read: “RESET” TO

   CALIBRATE
9. Push RESET

   Display will read: RESTORE BACKUP

   - = NO
10. Push - /CRSR

    Display will read: ZERO GAS

    RESET WHEN READY

11. In an area known to be free of organic vapors push RESET button

Display reads: MODEL 580
ZEROING

The display screen switches to SPAN GAS
RESET WHEN READY

12. Connect instrument probe to Tedlar bag filled with the calibration gas, then push RESET

    Display indicates: MODEL 580
    CALIBRATING

    Then the screen changes to: "RESET" TO
    CALIBRATE

13. Disconnect the calibration gas.
14. Push MODE/STORE to get out of the calibration menu.
15. OVM should read near 100 PPM for isobutylene. If benzene was used then it should read 60 PPM (response factor of 0.6)
16. ******IMPORTANT**! -Record the calibration value in the instrument log book***

9.0 Condensed Instructions for Calibration and Operation of the Thermo PDR 1000 Aerosol/Particulate Monitor - Tool Watch #212602 -04

9.1 Specifications

- Concentration measurement range (auto-ranging)[1]: 0.001 to 400 mg/m^3
- Scattering coefficient range: 1.5 x 10^{-6} to 0.6 m^{-1} (approx.) @ λ =880 nm
- Precision/repeatability over 30 days (2-sigma)[2]:
  ± 2% of reading or ±0.005 mg/m^3, whichever is larger, for 1-sec. averaging time
  ±0.5% of reading or ±0.0015 mg/m^3, whichever is larger, for 10-sec. averaging time
  ±0.2% of reading or ±0.0005 mg/m^3, whichever is larger, for 60-sec. averaging time
- Accuracy[1]: ±5% of reading ±precision
- Resolution: 0.1% of reading or 0.001 mg/m^3, whichever is larger
- Particle size range of maximum response: 0.1 to 10 µm
- Flow rate range (model *p*DR-1200 only): 1 to 10 liters/minute (external pump required)
- Aerodynamic particle sizing range (model *p*DR-1200 only): 1.0 to 10 µm
- Concentration display updating interval: 1 second
- Concentration display averaging time[3]: 1 to 60 seconds
- Alarm level adjustment range[3]: selectable over entire measurement range
- Alarm averaging time[3]: real-time (1 to 60 seconds), or STEL (15 minutes)
- Datalogging averaging periods[3]: 1 second to 4 hours
- Total number of data points that can be logged in memory: 13,391
- Number of data tags (data sets): 99 (maximum)
- Logged data:
  - Each data point: average concentration, time/date, and data point number
  - Run summary: overall average and maximum concentrations, time/date of maximum, total number of logged points, start time/date, total elapsed time (run duration), STEL concentration and time/date of occurrence, averaging (logging) period, calibration factor, and tag number.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

9.2 Handling Instructions

The *personal*DataRAM is a sophisticated optical/electronic instrument and should be handled accordingly. Although the *personal*DataRAM is very rugged, it should not be subjected to excessive shock, vibration, temperature or humidity. As a practical guideline, the *personal*DataRAM should be handled with the same care as a portable CD player.

If the *personal*DataRAM has been exposed to low temperatures (e.g. in the trunk of a car during winter) for more than a few minutes, care should be taken to allow the instrument to return near room temperature before operating it indoors. This is advisable because water vapor may condense on the interior surfaces of the *personal*DataRAM causing temporary malfunction or erroneous readings. Once the instrument warms up to near room temperature, such condensation will have evaporated. If the *personal*DataRAM becomes wet (e.g. due to exposure to water sprays, rain, etc.), allow the unit to dry thoroughly before operating.

Whenever the *personal*DataRAM is shipped care should be taken in placing it in its carrying case and repackaging it with the original cardboard box with the factory provided padding.

9.3 Safety Instructions

- Read and understand all instructions in this manual.
- Do not attempt to disassemble the instrument. If maintenance is required, return unit to the factory for qualified service.
- The *personal*DataRAM should be operated only from the type of power sources described in this manual.
- When replacing the internal 9V battery, follow the instructions provided on the back panel of the unit.
- Shut off *personal*DataRAM and any external devices (e.g. PC or Laptop) before connecting or disconnecting them.
- Shut off *personal*DataRAM before replacing the internal battery, or when plugging in or disconnecting the AC power supply or the optional rechargeable battery pack.

9.4 Handling and Operation

9.4.1 Model *p*DR-1000AN

The model *p*DR-1000AN can be operated in any position or orientation. Exposure to high intensity fluctuating light of the interior of the sensing chamber, through the front and back slotted air openings (see Section 5.5), should be avoided. Such large intensity transients may cause erroneous readings. Direct access of sunlight to the sensing chamber should be prevented.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Front Panel

Refer to Figures 1 (for model *p*DR-1000AN) or 2 (for model *p*DR-1200) for location of controls and display.

The front panel contains the four touch switches (keys) and the LCD screen required for the operation of the *personal*DataRAM.

The four touch switches provide tactile ("popping") feedback when properly actuated.

The ON/OFF key serves only to turn on the unit (while it is in the off state), and to turn it off (when it is operating).

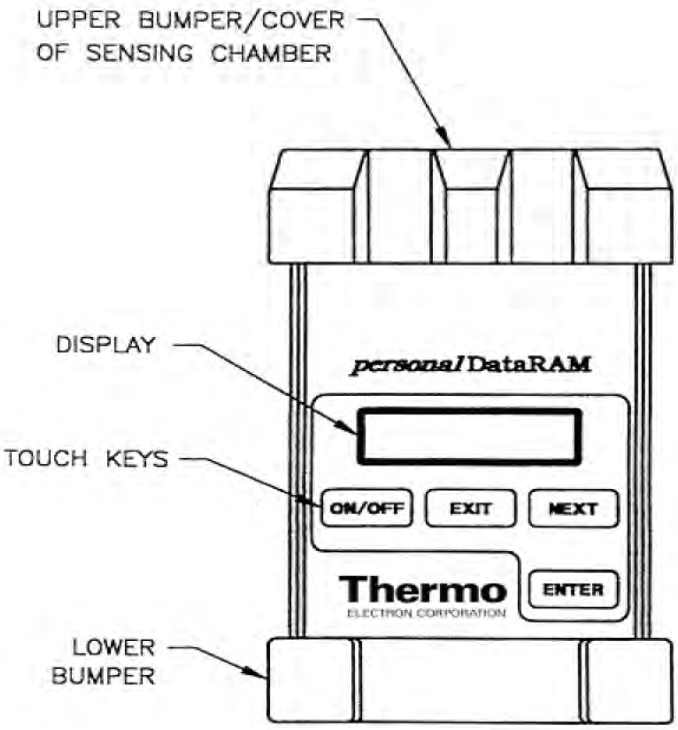


Figure 1 – FRONT PANEL (MODEL pDR–1000AN)

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Battery Installation

When shipped from the factory, the *personal*DataRAM will arrive without its replaceable 9V battery installed. Two fresh alkaline batteries (Duracell® type MN1604) are factory packed separately in the carrying case, one of which should be installed in the *personal*DataRAM when preparing it for operation.

NOTE: Whenever the *personal*DataRAM is to be left unused for an extended time (i.e. longer than a month), the 9V battery should be removed from the unit.

Removing the battery will lose neither the program, time/date keeping, nor stored data.

To install the battery proceed as follows:

- Hold the *personal*DataRAM upside down.
- Loosen thumbscrew that secures the battery compartment cover (see Figure 3 or 4), and remove that cover.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

- Observe battery polarity and the back panel battery orientation pattern (the negative battery terminal is the one closer to the side of the instrument).
- Insert the battery by sliding it in until it bottoms out. It should protrude slightly above the bottom surface of the instrument.
- Place battery compartment cover over battery and, while pushing down the cover firmly (taking care that the cover seats flush on the bottom surface of the *personal*DataRAM), tighten thumbscrew securely.

Battery Replacement

Normally, only a 9V Duracell® type MN1604 alkaline batteries should be used with the *personal*DataRAM in accordance the MSHA intrinsic safety approval.

Only fresh batteries should be used in order to ensure the maximum operating time. The *personal*DataRAM shuts itself off whenever the battery voltage falls below 6 volts (while retaining all programming and data). A fresh 9V alkaline battery, at room temperature, should provide typically 20 hours of continuous operation (please note that not all manufacturers produce batteries of equal capacity). Intermittent operation should extend the total running time because of partial battery recovery effects.

The approximate remaining battery capacity is indicated by the *personal*DataRAM (see Section 8.2) in increments of 1%, starting from 99%. If the remaining battery capacity is 40% or less, immediate restarting after shut off is automatically inhibited to prevent incomplete runs. If, nevertheless, a new run is to be initiated with low remaining battery capacity, do not shut off the *personal*DataRAM at the end of the previous run (i.e., remain in the Ready Mode, see section 7.0).

When significantly extended operating times are required (beyond the typical 20 hours), the use of either lithium or zinc-air batteries can be considered. The use of such alternative battery types can provide about 2 to 3 times longer operation than alkaline batteries.

AC Power Supply

A universal line voltage AC to DC power supply (Thermo Electron model *p*DR-AC) is provided as standard accessory with the *personal*DataRAM. This power supply can be used with any line with a voltage between 100 and 240 VAC (50 to 60 Hz). When using that power supply, its output plug should be inserted into the external DC receptacle at the base of the *personal*DataRAM (see Figure 3 or 4). Insertion of that connector automatically disables the internal 9V battery of the instrument. Removal of the *p*DR-AC plug from the instrument automatically re-connects the internal 9V battery.

NOTE: Before plugging in or unplugging the external power supply, the *personal*DataRAM must be shut off.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Zeroing the Model *p*DR-1000AN

Zeroing of the model *p*DR-1000AN requires a particle-free environment such as a clean room, clean bench, duct or area directly downstream of a HEPA filter, or the *p*DR-1000AN Z-Pouch (standard accessory). In some cases, a very clean, well air-conditioned office may offer a sufficiently low particle concentration environment

(i.e., ≤ 5 μg/m^3) for zeroing, as determined by another monitor (e.g., Thermo Electron DataRAM 4).

To zero the model *p*DR-1000AN by means of its Z-Pouch, proceed as follows:

- Wipe the outside surfaces of the *p*DR-1000AN to remove as much dust from those surfaces as possible before placing the instrument inside the Z-Pouch.
- In a reasonably clean environment, open the zipper of the Z-Pouch and place the *p*DR-1000AN inside it. Close the zipper shut.
- Open the small nipple on the Z-Pouch, and insert the fitting of the hand pump/in-line filter unit into the nipple.
- Start pumping the hand-pump until the Z-Pouch begins to bulge, and proceed with the steps in Section 8.1, pressing the keys of the instrument through the wall of the Z-Pouch. Then slowly continue to pump to maintain positive pressure within the Z-Pouch.
- After completing the zeroing (step 2. of Section 8.1) procedure, open the Z-Pouch zipper and remove the *p*DR-1000AN. Close the zipper and flatten the Z-Pouch while plugging its nipple, in order to prevent dust contamination of the interior of the Z-Pouch.
- The *p*DR-1000AN is now zeroed and ready for a measurement run.

To provide the particle-free air required to zero the *p*DR-1200, either of two methods can be used: a) place the instrument on a clean-air bench or in a clean room, or b) connect to the cyclone inlet the green zeroing filter cartridge supplied with the *p*DR-1200. In either case, proceed as follows:

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTIUNED INSTRUCTIONS

9.5 Abbreviated Instructions

9.5.1 Run Start/Stop

To power-up and start a measurement run without zeroing and without logging, proceed as follows:

- Key sequentially **ON/OFF, NEXT** and **ENTER**.

To terminate run and shut down, proceed as follows starting from the concentration screen (otherwise key **EXIT** first):

- Key sequentially **EXIT**, **ENTER**, **ON/OFF** and **ENTER**.

9.5.2 Resetting Procedure

The *personal*DataRAM memory can be reset through commands entered on its own keypad (i.e. without requiring a PC).

Resetting accomplishes the following:

- Erases all stored data from memory;
- Resets all parameters and operating conditions to their default values and conditions; and
- Cancels the zero correction offset.

WARNING: THE RESET TEST WILL ERASE ALL DATA STORED IN MEMORY AND SET ALL PARAMETERS TO FACTORY DEFAULT SETTINGS. DOWNLOAD ANY DATA BEFORE THE RESET PROCEDURE.

The procedure to reset the instrument is as follows:

Starting with the unit shut off, press the **EXIT** and **ENTER** keys at the same time, and while holding down those two keys, press **ON/OFF**. The screen will then indicate: PDR SELF-TEST...
and several diagnostic screens will appear in rapid sequence (see Section 16.0, Resetting/Electronics Checking Mode), ending in the message TESTING COMPLETE. The unit will shut off. When turned on again, the *personal*DataRAM memory will have been reset, as described above.

The **default** values and operating conditions of the *personal*DataRAM are:

- Logging period (LOG INTRVL): 60 seconds
- Logging status: disabled (LOGGING DISABLED)
- Alarm level: 1 mg/m^3

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

9.6 Operation

9.6.1 Start-Up

	KEY	DISPLAY	NOTES
1.	**ON/OFF**	START ZERO:ENTER GO TO RUN: NEXT	Before starting a run with the *personal*DataRAM, zero it (see Section 6.5) and key **ENTER** while the unit is exposed to particle-free air. Alternatively, key **NEXT** to go to RUN/READY mode. If **ENTER** is keyed:
2.	**ENTER**	ZEROING V2.00	Keep clean air flowing while ZEROING is displayed* for 1.1 min., followed by one of these screens:
		CALIBRATION: OK	or,
		BACKGROUND HIGH	or,
		MALFUNCTION	If CALIBRATION: OK, then go to step 3. If one of the other two screens is displayed, consult Section 12.0.
3.	**NEXT**	START RUN: ENTER READY: NEXT	To start a measurement run key **ENTER** (Section 8.3, step 1). To set up for a run and scroll logging/operating parameters, key **NEXT** (see Section 8.2).
4.	**ON/OFF**	TURN OFF PDR? Y:ENTER N:NEXT	Keying **ON/OFF** while the unit is operating will elicit this message to prevent accidental shut off. To confirm shut down, key **ENTER**. To continue operation, key **NEXT**.

*The number following the V on the screen refers to the installed firmware version.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

9.6.2 Setting Up for a Run (Ready Mode)

	KEY	DISPLAY	NOTES
1.	**NEXT**	LOGGING DISABLED	This screen indicates the logging status. To enable the logging function, key **ENTER**. Toggling of the on/off logging status can be done by keying ENTER.
2.	**ENTER**	LOG INTRVL 600s TAG#: 4	This message indicates that logging is enabled. Example is for 10-min log period, selected through the PC (see Section 9.0), and next free tag is #4.
3.	**NEXT**	ALARM: OFF	This screen indicates the alarm status. Keying **ENTER** repeatedly toggles through the 3 alarm modes:
4.	**ENTER**	ALARM: INSTANT LEVEL:1.50 mg/m^3	This enables the alarm based on the real-time concentration. The level (e.g. 1.50 mg/m^3) must be set on the PC.
5.	**ENTER**	ALARM: STEL LEVEL:0.50 mg/m^3	This enables the alarm based on the 15-min STEL value. The level (e.g. 0.50 mg/m^3) must be set on the PC.
6.	**NEXT**	ANALOG OUTPUT: DISABLED	This screen indicates the analog signal output status. Keying ENTER will enable the analog output. Toggling the analog output on/off can be done by keying **ENTER**:
7.	**ENTER**	ANALOG OUTPUT: 0 – 0.400 mg/m^3	This enables the analog output. The concentration range (e.g., 0 – 0.400 mg/m^3) must be set on the PC.
8.	**NEXT**	CAL FACTOR: 1.00 DIS AVG TIME 10s	This screen displays the calibration factor and the display averaging time. Edit via PC

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

9.	**NEXT**	BATTERY LEFT 83% MEMORY LEFT 96%	This screen displays the remaining battery charge, and the remaining percentage of free memory.
10.	**NEXT**	CONNECT TO PC	When this screen has been selected, the operating parameters can be edited and/or the logged data can be downloaded via the PC (see Section 9.0). If **NEXT** is keyed again, the screen returns to RUN/READY:
11.	**NEXT**	START RUN: ENTER READY: NEXT	The instrument is now ready to run following the procedure in section 8.3.

9.6.3 Measurement Run Procedure

KEY		DISPLAY	NOTES
1.	**ENTER**	LOGGING DISABLED	or, if logging was enabled:
		LOG INTRVL 600s TAG #: 4	Logging status will be displayed for 3 seconds.
		CONC*0.047 mg/m^3 TWA 0.039 mg/m^3	After a 3-second delay, the concentration screen appears values shown here are examples). CONC is the real-time and TWA is the time-averaged concentration. The "*" appears only if logging has been enabled.
2.	**EXIT**	TERMINATE RUN? Y:ENTER N:EXIT	To terminate the current run and return to the Ready Mode, key **ENTER**. To continue the run, key **EXIT**.
3.	EXIT	CONC*0.047 mg/m^3 TWA 0.039 mg/m^3	Keying **NEXT** successively scrolls the display to show various run values (elapsed run time, maximum, STEL, etc.). Keying **EXIT** returns to the concentration display.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

4.	**NEXT**	ET 06:12:49 ST 08:18:26MAY15	This screen shows the elapsed run time (ET) and the run start time/date (ST).
5.	**NEXT**	MAX: 0.113 mg/m^3 T 10:08:44 MAY15	This screen shows the maximum concentration of current run and time/date of occurrence.
6.	**NEXT**	STEL:0.058 mg/m^3 T 09:59:22 MAY15	This screen shows the 15-min STEL value of the current run and the time/date of occurrence.
7.	**NEXT**	BATTERY LEFT 83%	or, if logging was enabled:
		BATTERY LEFT 83% MEMORY LEFT 96%	This screen shows the amount of usable charge left in the battery and, if logging has been enabled, the overall amount of free memory left.
8.	**NEXT**	ANALOG OUTPUT: 0 – 0.400 mg/m^3	This screen shows the status of the analog signal output, and the range, if this output has been enabled.
9.	**NEXT**	CONC*0.047 mg/m^3 TWA 0.039 mg/m^3	The last **NEXT** command returns the display to the concentration screen.
10.	**EXIT**	TERMINATE RUN? Y:ENTER N:NEXT	As indicated in step 2, to end current run, key **ENTER**, to return to the Ready Mode:
11.	**ENTER**	START RUN: ENTER READY: NEXT	This keystroke terminates the current run and returns the unit to the Ready Mode.

If during a run the instrument memory is filled completely, or if all 99 tags have been used, the run is automatically terminated and the display will indicate:

RUN TERMINATED
FULL MEMORY

If a new run is initiated after the memory has been filled, the *personal*DataRAM can be operated only as a monitor without logging. The memory must then be cleared (see Section 7.3.2) first before logging can be enabled again.

10.0 Condensed Operating Instructions for the Use of the Lumex Mercury Vapor Analyzer - Tool Watch # 215501

10.1 Safety Guidelines

Safety guidelines

Important safety precautions

Read these rules completely before starting operation with analyzer.

- Carefully study all the sections of this operation manual, analyzer design and control functions.
- To avoid electrical shock, never work with the analyzer covers taken off.
- Charge the battery on a timely basis. Store the unit with the battery fully charged.
- Do not put extraneous objects inside the analyzer through its ports.
- Do not allow the ingress of liquids on the case or inside the analyzer.
- Use only the power supply which is provided.
- Never use a defective power supply cable, do not put any objects on the power supply cable, and locate it in a manner to avoid a trip hazard.
- Do not try to repair the analyzer or adjust its optical units and electronic boards by yourself, except for cases stipulated in troubleshooting section (Appendix).
- Call an authorized agent or certified service engineer in the following cases:
  - If the analyzer does not operate properly or its parameters have noticeably deteriorated.
  - If the analyzer has fallen down or if its case is damaged.
  - If a liquid has gotten inside the analyzer.
  - If you hear unusual sounds or sense unusual smell coming from the analyzer.
- Keep the analyzer at a minimum distance of 1 meter from heating devices and heat sources. Do not transport analyzer in the trunk of your car.
- Do not forget to switch the analyzer off when the working day is over and to disconnect the power supply unit from the mains if the analyzer is not used for more than 6 hours.
- Never work without absorption and dust filters.
- The analyzer should be switched off before any maintenance operations.
- When measuring mercury concentration, it is necessary to follow instructions and documents stating safety regulations for operation in chemical laboratories and safety rules for operation with electric appliances.

INSTRUCTIONS CONTINUED ON NEXT PAGE

10.2 Application

The RA-915 Light analyzer (base unit) is intended for measuring mercury vapor concentration in ambient air and in the air of residential quarters and production areas.

The analyzer can be used for on stream measuring of mercury vapor concentration in the air.

The analyzer is used for tackling environmental problems, technological processes monitoring, and for industrial sanitary and scientific research.

Basic analytical characteristics of the analyzer are given in the table below:

Minimum mercury vapor concentration in air ($\mu g/m^3$)	0.1
Maximum mercury vapor concentration under analysis ($\mu g/m^3$)	100

Main technical data and specifications

Built-it battery	6.3 V
External direct current source	10 – 14 V
A.C. power source, through external unit	220/110 V, 50/60 Hz
Power consumption	20 W
Dimensions (mm)	460 * 210 * 110
Weight (kg)	7.5

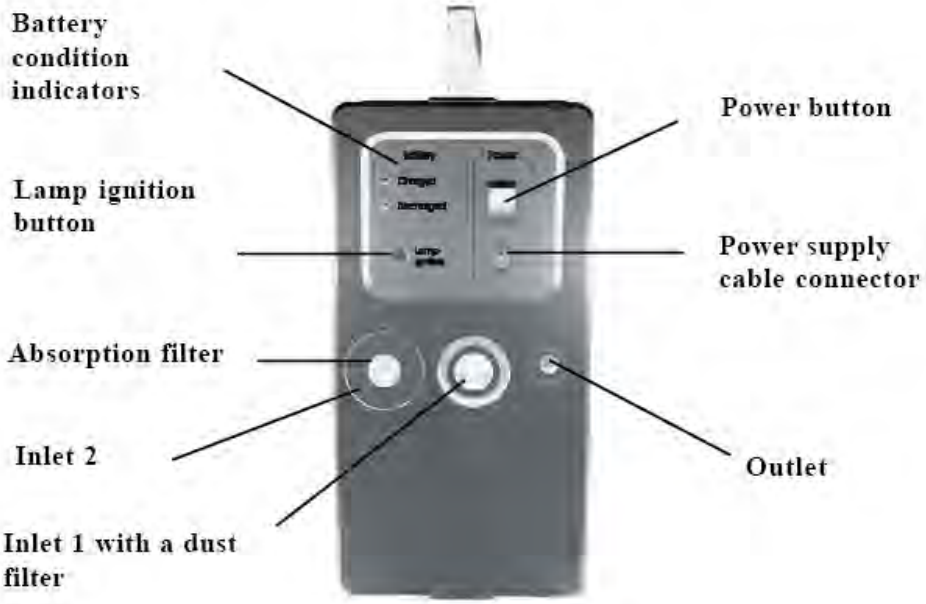
Operating conditions

Ambient air temperature	from +1 to 40 ^{0}C
Atmospheric pressure	84.0 – 106.7 kPa
Relative humidity	under 95 %, at 35 ^{0}C

INSTRUCTIONS CONTINUED ON NEXT PAGE

Appearance and functional controls

Front panel view



INSTRUCTIONS CONTINUED ON NEXT PAGE

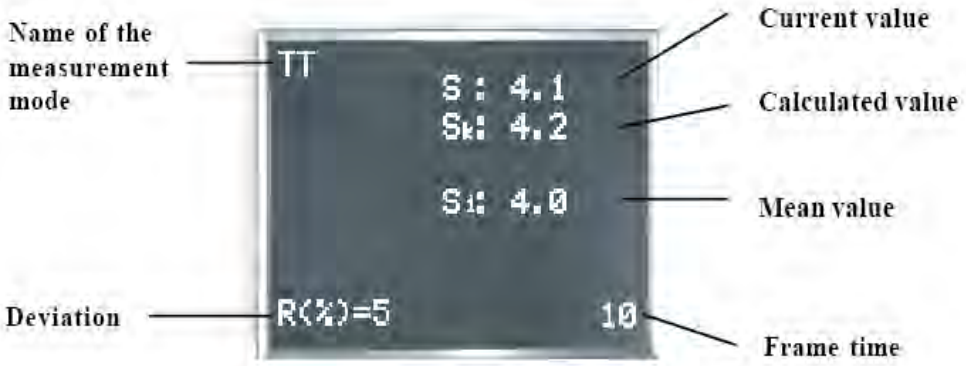
10.3 Test Instructions

The *TEST* command is intended for checking the analyzer serviceability.

1 Set the TEST handle (side panel of the unit) to the OFF position.
2 Select the TEST command on the display unit and press the **Ent** button.
3 The zero baseline will be established automatically over the next 25 s.
4 The ENTER THE TEST CELL message will be displayed.
5 Set the TEST handle of the test cell to the ON position
(after rotating it back and forth several times) and press the **Ent** button.
The window for the device serviceability check will appear.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS



Current value (S) is the mercury vapor concentration in the test cell at the current moment (it is displayed at a rate of 1 Hz).

Calculated value (S_k) is the calculated mercury vapor concentration in the test cell, which depends on the temperature of the test cell.

Mean value (S_i) is the mean mercury vapor concentration determined during the accumulation time.

Frame time is the countdown of the accumulation time in seconds (**frame time**, the **Parameters** command).

Name of the measurement mode (TT) - serviceability check-up.

Deviation (R, %) is the relative deviation of the measured value of the mercury vapor concentration in the test cell from the calculated value. It is consecutively displayed and retained during the frame time.

*The **Temperature** message may appear during the analyzer service-ability check. This indicates that the temperature of the test cell falls out of the allowable temperature range.*

6 If relative deviation **R is equal to, or less than 25 %**, the device is ready for operation and you may proceed.

7 Press the **ESC** button. The REMOVE THE TEST CELL message will appear.

8 Set the TEST handle of the test cell to the OFF position and press the **Ent** button. The MAIN MENU will be displayed.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

ON STREAM

The *ON STREAM* command is used for measuring the mercury vapor concentration in the air.

1 Set the TEST handle to the OFF position.
2 Set suitable parameters using the PARAMETERS command in accordance with the type of analysis.
3 Select the ON STREAM mode from the MAIN MENU and press the **Ent** button. The zero baseline will be established automatically over the next 25 s.

To determine the mercury vapor concentration in the ambient air in the ON STREAM mode, default parameters are sufficient.

4 On completion of the zero-signal measurement, the window for operation in the *ON STREAM* mode will appear.

Current value (S) is the mercury vapor concentration in the pumped air at the current moment. (It is displayed at a repetition rate of 1 Hz and is measured in ug/m³).

Mean value (S_i) is the mean mercury vapor concentration determined during the accumulation time. (It is displayed once per the accumulation time and is measured in µg/m³).

Frame time is the countdown of the accumulation time in seconds (**frame time**, the **Parameters** command).

Name of the measurement mode (SM) – the ON STREAM mode.

The compressor is switched on when the up arrow button is pressed at the display unit. The compressor is switched off when the down arrow button is pressed at the display unit. Switching off the compressor during pauses in operation extends battery life.

INSTRUCTIONS CONTINUED ON NEXT PAGE

CONTINUED INSTRUCTIONS

Measurement units. Mercury vapor concentration is measured in terms of $\mu g/m^3$.

1 $\mu g/m^3$ = 0.001 mg/m^3
1 $\mu g/m^3$ = 1 000 ng/m^3

*The **ALARM** warning may appear during operation in the **SM** mode. This means that the mercury vapor concentration in the air exceeds the preset **ALARM limit (AL)**.*

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11.0 Condensed Operating Instructions for the Use of the MSA Hydrogen Sulfide Meter - Tool Watch # 213501-05

Monitoring Capability			
Sensor Types	Instrument shall be available with the following gas sensing options:		
	Gas Type	Range	Resolution
	Oxygen	0-25%	0.1%
	Carbon Monoxide	0-500 ppm	1 ppm
	Hydrogen Sulfide	0-200 ppm	1 ppm

Basic Operational Features	
Instrument Turn-on	Button to turn instrument on must be clearly marked.
Inadvertent Shutoff	The instrument must be designed to protect against accidental shut off.
Zero Adjustments	The instrument shall provide a Fresh Air Setup (FAS) function at the user's discretion.
Zero Adjustment Safety Lockout	The FAS function will prevent users from zeroing out hazardous readings.
"Instrument On" Indicator	The instrument shall be provided with a periodic heartbeat signal indicating the instrument is in operation.

INSTRUCTIONS CONTINUED ON NEXT PAGE
CONTINUED INSTRUCTIONS

Instrument Alarms	
Visual Alarms	Visual alarms shall consist of bright flashing LED's and a positive indication on the display as to which gas sensor is in alarm.
Audible Alarm	The audible alarm shall be rated at 95 dB.
Vibrating alarm	The unit shall have an option to be equipped with a vibrating alarm.
Oxygen Alarms	The oxygen channel will have alarm setpoints for both oxygen deficiency and oxygen enrichment.
Alarms Setpoints	Alarm set points must be user settable.
STEL and TWA alarm	The instrument shall provide an audible alarm if the STEL or TWA levels are exceeded. The user will be able to select alarm setpoints for STEL and TWA (toxic channels only).
Power Alarms	The monitor will provide a minimum of 5 minutes warning to user of battery power loss in all environmental conditions. • Power alarms shall be both audible and visually indicated on display

Instrument Power	
Battery life indication.	The monitor shall provide the user with a "gas gauge" depicting estimated remaining battery operation time. • Battery gas gauge must always be visible when the instrument is turned on.

Calibration	
Calibration Tools	The unit shall require no special tools for calibration other than cylinder, regulator and tubing to supply gas to instrument.
Pushbutton Calibration	Calibration must be easily accomplished utilizing push buttons on the face of the instrument. Internal instrument access or tools shall not be necessary for calibration.
One-button Calibration	Instrument shall be capable of being calibrated to known gas concentrations with push of a single button.
Low Cost Calibration Kit	The instrument shall be available with an optional low cost gas testing kit to verify performance in field. This kit shall operate with a trigger-type aerosol canister and shall be capable of checking the performance of the standard instrument.

Certifications	
Intrinsic Safety Approval	The detector must be approved by : • U. S. and Canadian Nationally Recognized Testing Laboratories as intrinsically safe to Class I, Division 1, Groups A B, C and D. T code T4, T_{amb}= -20 to 50^0C • European Testing Laboratory as ATEX II 1 G EEx ia IIC T4 +50C T code T4, T_{amb}= -20 to 50^0C • European EMC complies to EN50270
Manufacturing System Quality Approvals	The instrument manufacturer must be certified compliant with ISO 9001 provisions.

Environmental	
Temperature	Normal Operation: -20 to 50° C H2S -40 to -20° C for short (15 minute) periods
Humidity	15-90% RH (non condensing) continuous 5-95% RH (non condensing) for short periods

12.0 Instructions for the use of the Handy Heat Index Checker (WBGT) 8758 – Tool Watch #983801

Operation Manual
Handy Heat Index Checker 8758

Features
1. Heat stress index(WBGT), temperature, humidity in one handy meter
2. Audible alarm for thermal condition monitoring
3. Sensor protecting cover
4. Tripod mountable
5. Connect to PC for data analysis

Specifications
Air temperature(TA) range: 0~50C(122F)
Air temperature accuracy: +/-0.6C
Globe temperature(TG) range: 0~80C(176F)
Globe temperature accuracy:+/-2C
WBGT Temp range: 0~50C(122F)
WBGT Temp accuracy: +/-2C
RH% range: 0~100% RH
RH accuracy: +/-3% RH (10~90% RH)
+/-5% RH for other range
Resolution: 0.1C/F, 0.1% RH
Battery life: > 1000 hours
Black ball size: Dia. 40mm, height 35mm
Meter size: 29.4(H)x 48.7(W)x 254mm (L)

Display and Indicators

1

1. WBGT(Wet Bulb Globe Temperature)
   A commonly used heat stress indicator that considers the effects of temperature, humidity and radiant energy.
2. TG(Black Globe Temperature)
   Measured by the black globe thermometer, which monitors the effects of direct solar radiation on an exposed surface.
3. TA: Air Temperature
4. RH%: Relative Humidity
5. IN/OUT:
   Represent the measuring environment that is with or without direct sun exposure and display corresponding WBGT values using the following equations:
   Indoor/outdoor no sun: WBGT=0.7 WB + 0.3 TG
   Outdoor or full sun: WBGT=0.7 WB + 0.2 TG + 0.1 TA

Function Keys
Φ/SET: Turn on and off the meter
Enter alarm setting mode
NEXT: Select digit in setting mode
Exit from setting mode
MODE/▲: Change displaying mode
Shift the IN/OUT display
Increase value in setting mode
Φ/SET+ MODE/▲: Set as non-sleep mode
MODE/▲+NEXT: Select C or F
Φ/SET+NEXT+MODE/▲: Enter RH calibration mode

Operating Instructions
1. Power on/off
   PressΦ/SET to turn on and turn off the meter. When meter is power on, all indicators are shown on the LCD for some seconds and then enter measuring mode.
2. Measurement
   Pull down the protecting cover of sensor before taking measurements.

2

2.1 Select displaying mode
The meter measures TG(Black Globe Temperature), TA(Air Temperature), RH(Relative Humidity), and calculate the WBGT index as guides of human thermal comfort. Press MODE/▲ button to select the measurement display.

2.2 Switch IN/OUT measurement
The meter measures WBGT index with or without direct sun exposure.
Users can shift the IN/OUT display by pressing MODE/▲ for more than 1 second in measuring mode.

2.3 Select temperature Unit
To select C or F as the preferred temperature unit, press MODE/▲and NEXT simultaneously in measuring mode.

3. Alarm setting
The meter features audible alarm to give warnings of possible thermal stress. Users can adjust the alarm value as r ecommended in different fields by pressing Φ/SET button for 2 seconds while power off. In setting mode, press NEXT to select digit and MODE/▲ to increase the value. The setting range is 20.0~37.2C(68.0~99.0F) and "OUT" will show to indicate out of range value. When the alarm value is set, press NEXT for 2 seconds to enter measuring mode.

4 Low battery indication
Low battery indicator shows on the lower side of LCD when the voltage gets low.

5 Auto power off
The meter is preset auto power off after 20 minutes non-operation. To set it non-sleep, pressΦ/SET and MODE/▲ simultaneously for 2 seconds when power off. It will display "n" and then enter measuring mode. Reset will be invalid when meter is turned off.

6. Data analysis
Connect the meter to PC via RS232 port for on-line logging, analysis or printing. The protocol is as follows. 9600 bps, 8 data bits, no parity.

3

RH Calibration
Unscrew the black ball and sensor shell while meter is power off. Plug the probe into 33% calibration salt, press,Φ/SET, NEXT, and MODE/▲ buttons simultaneously for 2 seconds to enter calibration mode. A value "3X.X%" is blinking during calibrating and stops when completed in around one hour. Then plug the probe in 75% calibration salt and press "MODE/▲" for 2 seconds to start 75% calibration. Wait an hour until "7X.X%" stops blinking then complete the calibration and back to measuring mode. The difference between the last display value and calibration salt should be within +/-0.3%, otherwise it is a failed calibration.

Calibration needs to be done under 23+/-2C air temperature.
Users can terminate the calibration by pressingΦ/SET.

Troubleshooting
1. Power on but no display
   a. Make sure to press Φ/SET more than 100mS.
   b. Check whether the batteries are in good contact and correct polarity.
   c. Replace with fresh battery or take out the batteries for one minute and put them back.
2. Display disappeared
   a. If low battery indicator showed before display disappeared, replace with new batteries.
   b. Disable Auto Power Off function for long time operation.
3. Calibration failure
   a. If low battery indicator showed before display disappeared, replace with new batteries.
   b. Check whether the probe is well plugged into the salt bottle and no air comes in.
   c. Check whether the air temperature is within 23+/-2C.
4. E2: The value is underflow.
5. E3: The value is overflow.
6. E4: The value is error because of original data.
7. E11: RH calibration error. Do the calibration again.
8. E33: Circuit error. Return the meter to the vendor for repair.

4

Bay West

Standard Operating Procedure

Sample Custody

CORP-ENV-004-1510208

Revised: February 10, 2012

Review and Approval:

Developed by: _____ Date: 02-17-2012
Signature
QA/QC Manager
Title

Reviewed by: _____ Date: 02-17-2012
Signature
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Approved by: _____ Date: 02/20/2012
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Vice President of Operations
Title

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

Due to the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are provided to the client or possibly introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in Bay West, Inc. (Bay West) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

2.0 DEFINITIONS

Sample - A material to be analyzed that is contained in single or multiple containers representing a unique location and possessing a unique sample identification number.

Sample Custody - A sample is under custody if:

1. It is in your possession;
2. It is in your view, after being in your possession;
3. It was in your possession and then locked up to prevent tampering; and/or
4. It is in a designated and identified secure area.

Chain-of-Custody Record - Form used to document the transfer of custody of samples from one individual to another.

Custody Seal - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

Sample Label - Adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag - Tag attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

3.0 RESPONSIBILITIES

Sampler - The Sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Site Supervisor - The site supervisor is responsible for ensuring that strict Chain-of-Custody procedures are maintained during all sampling events. The site supervisor is also responsible for coordinating with the subcontractor laboratory to ensure that adequate information is recorded on custody records.

4.0 REQUIRED EQUIPMENT

This section provides a list of equipment to be used but does not necessarily include all equipment such as sample containers and personal protection equipment. The following is a general list of equipment that should be obtained in the office prior to initiating field work:

- Chain-of-Custody Records (applicable Bay West forms);
- Custody seals;
- Sample labels or tags; and
- Clear Tape.

5.0 PROCEDURES

5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a Chain-of-Custody Record. This procedure will be followed for all samples collected or split samples accepted.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual fieldwork. As few people as possible should handle samples.
2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
3. Sample labels or tags shall be completed for each sample, using waterproof ink.

Transfer of Custody and Shipment

1. Samples are accompanied by a Chain-of-Custody Record (see Figure 1 for example of Chain-of-Custody Record). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the

time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the appropriate laboratory.

- The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g. Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the form; however, the airbill tracking number should be recorded on the Chain-of Custody.
- In all cases, it must be readily apparent that the person who initially received custody is the same person who relinquished custody to the next custodian.
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the Chain-of-Custody Record.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each cooler or shipment unit. When shipping multiple coolers containing samples from the same project, indicate on the Chain-of-Custody that the Cooler/Record is 1 of 2 or 1 of 3, etc. to reflect the total number of coolers and Chain-of-Custody records shipped.
3. All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and the copies will be retained by the site supervisor and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the site supervisor as part of the permanent documentation.

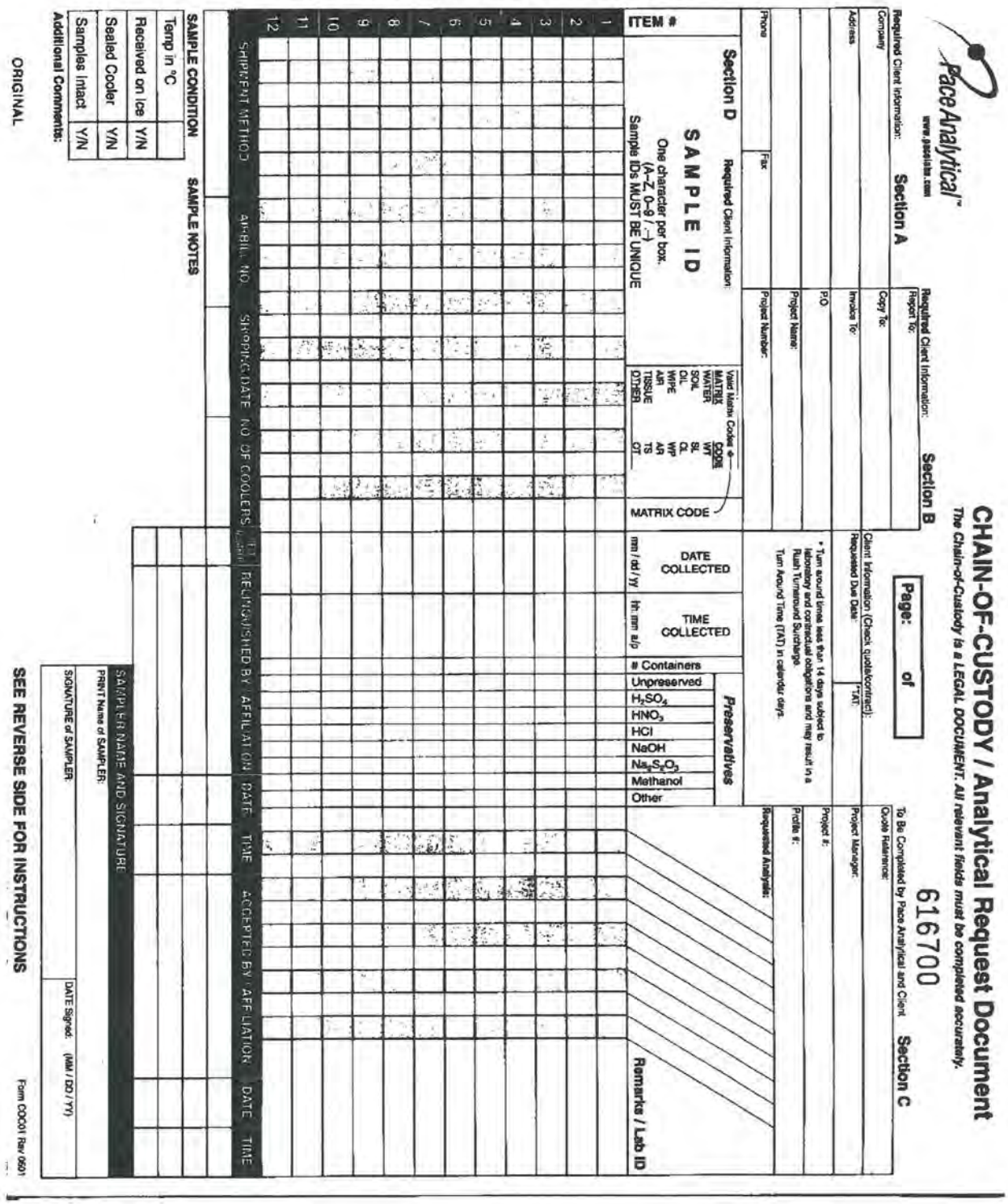
Procedure for Completing Bay West Chain-of-Custody Record
(Refer to attached Figure 1)

1. Record project number.
2. Record sampler for the project.
3. Record the name and address of the laboratory (include both the Bay West Project Manager and the laboratory Project Manager name) to which samples are being shipped.
4. Record the unique Chain-of-Custody record number for each Chain-of-Custody used in shipping for the day. (Note: not all CoCs have record numbers)
5. Enter the project name/location code or number.
6. Record overnight courier's sample airbill tracking number on the Chain-of-Custody.
7. Note sample type (matrix) and matrix code. Include the matrix code on the Chain-of-Custody Record.
8. Record sample identification number.
9. Enter date of sample collection.

10. Enter time of sample collection in 24-hour time format.
11. List parameters for analysis and the number of containers submitted for each analysis.
12. Enter MS/MSD (matrix spike/matrix spike duplicate) if sample is for laboratory quality control, or other remarks (e.g. sample depth).
13. Record the type of the preservative used to preserve the sample. Use the remarks column if no space identification dedicated to preservative.
14. All samplers must sign in the space provided.
15. The originator verifies (checks) the information entered on the Chain-of-Custody and then signs the "Relinquished by" box, prints his/her name, and enters the current date and time (24-hour). Edits to the Chain-of-Custody should be made by drawing a single line through the incorrect information, recording the correct information, and initialing and dating next to the change.
16. Upon completion of the custody record form, the top two copies (usually white and yellow) shall be sent with the samples to the laboratory; the bottom copy (usually pink) is retained for the project files. Additional copies will be retained for the project file or distributed as required to the appropriate sample coordinators. In all cases, the original (top) copy is sent to the laboratory.
17. The laboratory sample custodian receiving the samples checks the sample label information against the custody record form. He or she also checks sample condition and notes anything unusual under "Description/Comments" on the custody record form. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the appropriate copy. The original copy is returned to Bay West.

NOTE: Different clients, have different *styles* of Chain-of-Custody records. In all cases, the above listed information must be included on all Chain-of-Custodies.

18. Figure 1
EXAMPLE PACE Chain-of-Custody Record



Sample labels or tags will be utilized for all samples collected or accepted for Bay West projects.

1. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.
2. Sample tags will be securely attached to the sample bottle. On 80 oz. amber bottles, the tag string may be looped through the rink style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted and re-looped around the neck until the slack in the string is removed.
3. One label or tag will be completed for each sample container collected. Each label or tag will be completed as follows; labels are completed with the equivalent information:
   - Record the Project Code (i.e., project name or task number);
   - Enter the Station Number if applicable;
   - Record the date to indicate the month, day, year of sample collection;
   - Enter the time (24-hour) of sample collection;
   - Record the sample location;
   - Samplers must sign/initial in the space provided; and
   - Under "remarks" add additional, relevant information, including any preservative.

5.2 Custody Seals

Custody seals must be placed on the shipping containers prior to shipment. The seal should be signed and dated by a field team member.

Custody seals may also be placed on individual sample bottles, depending on project requirements. Check with the project manager or refer to EPA regional guidelines for direction.

5.3 Sample Shipping

Bay West's Standard Operating Procedure: Packaging and Shipping of Environmental Samples (CORP-ENV-006-15102006) establishes a uniform method for packaging and shipping low-level environmental samples.

Every effort should be made to ship samples on the day they are collected. In the event samples will not be shipped on the day of collection, proper preservation techniques must be maintained until the samples reach the laboratory. Sample bottles will not be

open to test for preservation (e.g. pH) as this may compromise the sample. However, maintaining the samples at proper temperature, 4°C ± 2°C, is critical to sample integrity. Sufficient ice should be on hand to maintain proper temperatures during and after sampling. Samples held for shipment the next day must be checked for proper temperatures prior to shipping. This check should be noted in the log book.

It is inadvisable to hold samples for more than one day prior to shipping to the lab. All samples have holding times associated with their individual analysis and delaying shipping may compromise the lab's ability to meet the holding time requirements. These requirements may be found in the project Field Sampling Plan or the QAPP.

If samples are shipped on a Friday for Saturday lab delivery, the laboratory must notifed in advance of their arrival. A phone call should be placed to: the lab's Project Manager, the Bay West Project Manager and/or the Project Chemist.

If particular questions arise regarding the shipping of a certain sample, the lab's Project Manager is an *excellent* resource on proper shipping technique.

6.0 DOCUMENTATION

All notes/comments associated with the sample custody will be recorded in a project specific field notebook in accordance with the Bay West SOP for Sample Custody.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

7.1 Restrictions/Limitations

For EPA Contract Laboratory Program (CLP) sampling events, combined chain-of-custody/traffic report forms or other EPA-specific records may be utilized. Refer to regional guidelines for completing these forms.

8.0 REFERENCES

U.S. Environmental Protection Agency, *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.

U.S. Environmental Protection Agency, *Sampler's Guide to the Contract Laboratory Program*, EPA/540/P-90/006, December 1990.

American Society for Testing and Materials, *Standard Guide for Chain-of-Custody Procedures*, ASTM D4840-99, 2010.

Bay West

Standard Operating Procedure

Classification and Description of Soil, Sediment, and Rock

CORP-ENV-007-1577802

Revised: October 2, 2012

Review and Approval:

Developed by: _____ Date: October 2, 2012
Signature

Manager, Environmental Division
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Reviewed by: _____ Date: October 2, 2012
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QA/QC Manager
Title

Approved by: _____ Date: October 2, 2012
Signature

Vice President of Operations
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TABLE OF CONTENTS

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the classification and description of unconsolidated material (soil, sediment) and rock samples collected during field operations performed by Bay West, Inc. (Bay West).

1.1 Purpose

The installation of monitoring wells, piezometers, boreholes, and test pit excavations are standard practice at many sites requiring environmental investigations. These activities require that a trained geologist or other earth scientist provide descriptions of subsurface material encountered. The purpose of this SOP is to serve as a guide for recording basic classification and descriptive information with emphasis on soil, sediment, or rock properties that affect ground water flow and contaminant transport. In order to make descriptions as uniform and consistent as possible, this SOP provides a list of references to be used in the field and a sequence for recording information on a standardized log form.

1.2 Scope

This SOP is applicable to all sites requiring a subsurface investigation, including sediment locations, by Bay West.

2.0 DEFINITIONS

The following definitions correspond to the list of descriptors outlined in **Section 5.2.1**. The definitions are provided to aid the field geologist in identifying the types of information and tools that are used to accurately describe a sample and record the description on soil boring log and coring log forms (**Attachment A**). Descriptive criteria and terminology presented are for visual classification and manual tests. Similar soils, sediment, or rocks may be grouped together; for example, identify one sample and describe it completely. Then identify others samples as similar based on performing only a few of the identification and description procedures. Descriptive information should be evaluated and reported on every sample.

2.1 Descriptors for Soil and Sediment

Naming Soils and Sediments. Use field equipment, reference charts, and American Society for Testing and Materials (ASTM) Standards presented in **Attachments B**, **C**, and **D** to name soils and sediments.

Texture. In examining soils and sediments, the texture shall refer to:

- Particle size, distribution, and range – provide percentage estimates (see the USCS in Attachment C).
- Angularity – angular, subangular, subrounded, rounded (see Attachment C and Table 1 and Figure 3 of Attachment D).
- Shape – flat, elongated.

Color. Color may be determined using the Munsell color chart. If the Munsell Color Chart is used, list the Munsell number that corresponds to the color. If the soil/sediment is mottled, list individual color names and provide an overall best color name. Accurate comparison is obtained by holding the sample directly behind the apertures separating the closest matching color chips (Munsell 1994).

Structure. There are several different sedimentary structures. Criteria for describing structure can be found in Table 7 of **Attachment D** and include stratified, laminated, fissured, slickensided, blocky, lensed, and homogeneous. Additional structure names are bedding, cross bedding, laminations, and burrows. Structures should only be included in the description if found in the samples.

Consistency. For intact fine-grained samples, describe the consistency in accordance with the clay consistency scale in **Attachment C** or Table 5 of **Attachment D**. Both tables present a scale from very soft to hard using a thumb penetration test. **Attachment C** also correlates the sample to the blow counts used in collecting hollow-stem auger samples.

Cementation. In some cases the sample may be cemented and should be described as weak, moderate, or strong cementation as described in Table 6 of **Attachment D**.

Moisture condition. Moisture condition refers to the amount of water within the sample. The soil/sediment should be described as dry, moist, or wet.

Odor. The purpose of many environmental investigations is to determine the nature and extent of contamination. Therefore, when dealing with potentially contaminated material, smelling for unusual odors is strongly prohibited. However, if an odor is apparent it should be noted on the soil boring log form. Possible descriptors are organic (decaying vegetation) or unusual odors (i.e., petroleum, chemical).

Evidence of Contamination. Examine the sample and note any obvious signs of contamination (e.g., streaking, free product, or discoloration). Note observations in the field book along with any readings from the photoionization or flame ionization detector (PID/FID). In addition, record PID/FID readings on the soil boring log form.

Other. Secondary features affecting porosity and permeability such as bioturbation are particularly important and should be described if observed. Particular attention is to be given to recording exact locations of water tables, perched saturated zones, and description of contaminants that may be visible (i.e., non-aqueous phase liquid).

2.2 Descriptors for Rock

Rock name. In naming sedimentary, igneous, and metamorphic rock, use field equipment to examine the specimen for mineralogy and use the appropriate classification charts. The first page of the American Geological Institute (AGI) Data Sheets for sedimentary, igneous, and metamorphic rock is included in **Attachment** E.

Use a simple descriptive name, sufficient to provide others with possible properties of the rock type. State the name and capitalize the first letter. If available, the name may include the stratigraphic unit name and/or geological age.

Lithology (composition/grain size/texture/color). Provide the following lithologic information:

- The mineralogical description (composition) of the rock;
- Grain shape and size or sizes;
- Porosity;
- Texture (vesicular, porphyritic, schistose);
- Contacts. Note any significant change in lithology (such as gradational contacts within sediments or sharp contacts such as sediments over rocks). Note if the contacts are erosional, gradational, or sharp and the depth below the ground surface; and
- Color. Color may be determined using the Munsell color chart and listing the Munsell number that corresponds to the color. Provide the wet color of fresh and weathered surfaces.

Discontinuities. These include shears, joints, fractures, cavities, secondary mineralization, or contacts. The rock types that may be encountered during drilling may have fractures or joints present within them. Note any observed fractures and describe the density of fractures. If cavities or vugs are present, note the density of the voids and estimate the size of the voids. If fractures or cavities contain evidence of secondary minerals such as zeolites, clays, or iron oxides, then describe the mineral fill.

Odor. The purpose of many environmental investigations is to determine the nature and extent of contamination. Therefore, when dealing with potentially contaminated material, smelling for unusual odors is strongly prohibited. However, if an odor is apparent it should be noted on the core log form. Possible descriptors are earthy or unusual odors (i.e., petroleum, chemical).

Evidence of Contamination. Examine the sample and note any obvious signs of contamination such as streaking, free product, or discoloration. Note these observations in the field book along with any readings from PID/FID. In addition, record PID/FID readings on the core log form.

Other. Secondary features affecting porosity and permeability such as fractures (joints or faults) or cavities should be described if observed. Particular attention is to be given to recording exact locations of water tables, perched saturated zones, and description of contaminants that may be visible (i.e., non-aqueous phase liquid). Also note coring loss/recovery, weathering/alteration, secondary mineralization, degree of induration, degree of vitrification for volcanic rocks, and hardness. Provide a description of thickness of bedding, banding, or foliation if present. Include an exact measurement of apparent bed thickness when logging core to supplement terminology such as “thin” or “thick.”

3.0 RESPONSIBILITIES

Field Geologist/Site Supervisor – The Field Geologist/Site Supervisor performing the classification and description of soil, sediment and rock samples is responsible for making a consistent and uniform log and for turning in field forms and logbooks to the Project Manager (PM).

Project Manager (PM) – The PM is responsible for maintaining logbooks and forms and for approving techniques of the classification and description of soil, sediment, and rock samples not specifically described in this SOP.

4.0 REQUIRED EQUIPMENT

The classification and description of soil, sediment, and rock samples requires a minor amount of field equipment for the geologist. This section provides a list of equipment to be used but does not include equipment such as drill rigs, PID/FID, sampling equipment, and personal protection equipment. The following is a general list of equipment that may be used:

- Field logbook;
- Soil boring log and coring log Forms (Attachment A);
- Clipboard;
- Plastic sheeting;
- Sampling trays;
- Waterproof pens;
- 10x magnifying hand lens;
- Reference manuals and field charts. These may include:
  - Standard Abbreviations and Sample Graphics (Attachment B);
  - Geotechnical Gauge (A black and white copy is included in Attachment C for reference. Use the original chart for field comparison);
  - Munsell Soil Color Chart (Munsell 1994);
  - ASTM Designation: D 2488-93, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) (ASTM 1993). A copy is included in Attachment D;
  - AGI Data Sheets for Geology in the Field, Laboratory, and the Office (AGI 1982). A copy of the first page of the field classification for Igneous, Sedimentary, and Metamorphic Rocks is included in Attachment E for reference. Use the original AGI Data Sheets for field comparison;
  - U.S. Department of Interior (DOI), Engineering Geology Field Manual Second Edition, Volume I (DOI 1998); and
  - Dictionary of Geologic Terms (AGI 1984).

5.0 PROCEDURES

The descriptive protocol presented here must be followed in making basic observations. Any further descriptions must follow a protocol that is published and generally recognized by the geologic community as a standard reference. However, if the user has expertise in a particular field of petrology or soil science that allows for descriptions of certain geologic sections beyond the basic level required by this SOP, descriptions may be expanded. This should be done only with approval of the PM.

This SOP is a guide for recording visual observations of samples in the field aided by a 10x hand lens and other simple tools. Field descriptions should be supplemented by petrographic analysis and sieve analysis when the PM needs data on numerical grain-size distributions, secondary porosity development, or other data that can be collected by these methods.

5.1 Field Preparation

This section provides a list of materials and equipment to be used, but does not necessarily include all equipment such as sample equipment, sample containers, and personal protection equipment. Obtain the following materials in the office prior to initiating field work:

- Obtain field logbook;
- Obtain soil boring and/or coring log form;
- Coordinate schedules/actions with PM;
- Obtain necessary field equipment (i.e., hand lens);
- Obtain Bay West reference field charts (i.e., Abbreviations and Sample Graphics, Geotechnical Gauge, Munsell Soil Color Chart, AGI Data Sheets, Engineering Geology Field Manual; Dictionary of Geologic Terms);
- Review field support documents (i.e., sampling plan, health and safety plan); and
- Review applicable geologic reference such as U.S. Department of Agriculture Soil Conservation Survey Soil Surveys and/or geologic maps and logs from previous site investigations.

5.2 Field Activities

This section provides field classification procedures for providing descriptions of soil, sediment and rock along with definitions of descriptors.

5.2.1 Procedures for Classification and Description of Soil, Sediment, and Rock

This section presents a list of descriptors to be used in the classification and description of soil, sediment, and rock. The descriptors are further defined in **Sections 2.1** and **2.2**. Similar samples may be grouped together; for example, one sample should be identified and described completely, with the others identified as similar based on performing only a few of the identification and description procedures. Descriptive information should be evaluated and reported on every sample (DOI 1998).

It is often more practical to use abbreviations for often-repeated terminology when recording descriptions. For the terms given in this SOP and Attachments, or the associated charts to be used for description in the field, use only the designated abbreviations (see **Attachment B** for examples obtained from **Attachments C** and **D**). Other abbreviations are allowed. However, the abbreviation must be defined and recorded on the soil boring and/or coring log form the first time it is used and must be recorded at least once for every soil boring and/or coring log form. Field geologists are cautioned to limit the use of abbreviations to avoid producing a log that is excessively cryptic.

5.2.1.1 Description of Soil and Sediment

Soil and Sediment comprises a significant portion of the sections of interest at Bay West sites. The shallow subsurface is very important to the hydrologic investigation, as this is the portion of the geologic section where infiltration first occurs. Much of the contamination at sites being investigated is surface contamination, and therefore lies on, or within, the upper portion of the surficial material.

Descriptors for soil and sediment shall be recorded in the sequence outlined below. Each description shall be separated by a comma. Interpretive comments shall be recorded separately in the field log book.

- Name (sand, silt, clay, etc.) (Local name if available).
- Texture (particle size, particle size range, angularity, shape).
- Color.
- Structure.
- Consistency.
- Cementation.
- Moisture condition.
- Odor ("smelling" the sample is not recommended. However, if an odor is apparent, it should be noted on the log form).
- Evidence of Contamination.
- Other (i.e., water level, roots, burrows, caving of trench or bore hole, composition of samples, HCL reaction).

Attachment F includes an example of a completed soil boring Form using this SOP to classify and describe soil and sediment samples.

5.2.1.2 Description of Rock

Sedimentary rocks consist of lithified detrital sediments such as sandstone and shale, chemically precipitated sediments such as limestone and gypsum, and biogenic material such as coal. Igneous rocks, volcanic and plutonic, and metamorphic rocks are not as commonly observed at work sites, but they may be found interspersed in the sedimentary section as ash layers and as bedrock. Where they form bedrock, the development of fractures and vugs is important to their hydrologic properties. If the

logger is unsure of the name of the rock because of difficulty in determining mineralogy, the name shall be accompanied by a question mark. The first page of the AGI Data Sheet classification scheme for naming sedimentary, igneous, and metamorphic rocks is included in Attachment E. The actual AGI Data Sheets (AGI 1982) or the Engineering Geology Field Manual (DOI 1998) should be used in the field to name rocks.

Descriptors for rock shall be recorded in the sequence outlined below. Each description shall be separated by a comma. Interpretive comments shall be recorded separately in the field log book.

- Rock name (local name if available);
- Lithology (composition/grain size/texture/color);
- Discontinuities;
- Odor;
- Evidence of contamination; and
- Other (i.e., coring loss/recovery, water level, weathering/alteration, secondary mineralization, bedding/foliation/flow texture, degree of induration, hardness).

Attachment F includes an example of a completed core log form using this SOP to classify and describe rock cores.

6.0 DOCUMENTATION

Individuals classifying and describing soil, sediment, and rock samples will record their observations in a commercially available, bound field logbook (e.g., Lietz books) and/or on individual soil boring and/or coring log forms. When using a bound field logbook, record the same data required on the soil boring and/or coring log form. Data from the field logbook must be transcribed to the soil boring and/or coring log form if filling in the form in the field is not feasible. However, the data must reflect the same information that is recorded in the field logbook. Any editing of field logbook data requires the erroneous information to be crossed out with a single line as not to obscure the initial entry, and the entry initialed and dated by the Field Geologist. The correct information will be entered immediately adjacent to the previous entry. In addition, if data are transcribed to the soil boring and/or coring log form, it should be done within one day of the original data recording. All blanks in the soil boring and/or coring log form must be filled out. Enter “NA” if an item is not applicable. The source of the reference material or field charts used must be recorded on the log form or in the field logbook

In addition to the information on the soil boring and/or coring log form, the Field Geologist shall fill in appropriate information into the logbook as described in the sampling plan and other pertinent information such as rig shut down, rig problems, failures to recover cores, etc.

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

7.1 Restrictions/Limitations

Only geologists or similarly qualified persons trained in the description of soil, sediment, and rock are qualified to perform the duties described in the SOP. The PM will have the authority to decide whether or not an individual is qualified for this task.

8.0 REFERENCES

American Geological Institute (AGI). (1984). *Dictionary of Geological Terms* (3rd Ed.). Anchor Press: New York, NY.

American Society for Testing and Materials (ASTM 1993). *Standard Practice for Description and Identification of Soils* (Visual-Manual Procedure), ASTM Designation: D 2488-93, November.

Dietrich, R. V., Dutro, J. T., & Foose, R. M. (1982). *American Geological Institute (AGI) Data Sheets for Geology in the Field, Laboratory, and Office* (2nd Ed.). American Geological Institute: Falls Church, VA.

DOI 1998, *Engineering Geology Field Manual (*2nd Ed.) Volume 1, Reprinted 2001.

Munsell Color, 1994 Revised Edition, *Munsell Soil Color Charts*: New Windsor, NY.

9.0 APPENDICES

Note: These Appendices are for informational purposes. Other equivalent charts or logs may be used. All materials used must be referenced accordingly.

ATTACHMENT A Bay West, Inc. Soil Boring Log and Coring Log Forms

ATTACHMENT B Standard Abbreviations and Sample Graphics for Soil, Sediment and Rock

ATTACHMENT C Geotechnical Gauge

ATTACHMENT D ASTM Designation: D2488-93

ATTACHMENT E First Page of the AGI Data Sheets on Sedimentary, Igneous and Metamorphic Rocks

ATTACHMENT F Example Completed Soil Boring Log and Completed Coring Log

ATTACHMENT A

Soil Boring Log and Coring Log Forms

Bay West

Project Name: _____
Project Number: _____
Driller: _____
Geologist: _____

Soil Boring No.: _____
Well No.: _____
Total Depth: _____
Drilling Method: _____
Sampling Method: _____
Grade Elevation: _____
Date Started/Completed: _____

Depth	Description - Remarks	Graphic Section	Munsell Color	USCS Class.	Analytical Sample	REC (in.)	Blows	Headspace PID/FID/ TLV
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

Bay West

Project Name: _____
Project Number: _____
Driller: _____
Geologist: _____

Rock Core No.: _____
Well No.: _____
Total Depth: _____
Drilling Method: _____
Sampling Method: _____
Grade Elevation: _____
Date Started/Completed: _____

Depth	Description - Remarks	Graphic Section	Munsell Color	Recovery/ loss (in.)	Headspace PID/FID/ TLV
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
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21					
22					
23					
24					
25					
26					
27					
28					
29					
30					

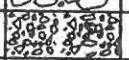
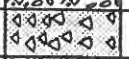
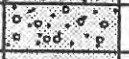
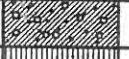
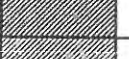
ATTACHMENT B

Standard Abbreviations and Sample Graphics for Sediment and Rock

Bay West

Project Name: Standard Abbreviations and Sample Graphics
Project Number:
Driller:
Geologist:

Soil Boring No.:
Well No.:
Total Depth:
Drilling Method:
Sampling Method:
Grade Elevation:
Date Started/Completed:

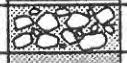
Depth	Description - Remarks (symbols)	Graphic Section	Munsell Color	USCS Class.	Analytical Sample	REC (in.)	Blows	Headspace PID/FID/ TLV
	Cobbles and Boulders							
1	Well graded gravel (GW)							
2	Poorly graded gravel (GP)							
3	Sandy well graded gravel [s(GW)]							
4	Silty gravel (GM)							
5	Clayey gravel (GC)							
6	Gravelly poorly graded sand [g(SP)]							
7	Well graded sand (SW)							
8	Poorly graded sand (SP)							
9	Silty sand (SM)							
10	Clayey sand (SC)							
11	Silt (ML)							
12	Sandy silt (ML/SM)							
13	Clayey silt (ML/CL)							
14	Lean clay (CL)							
15	Sandy clay (CL/SC)							
16	Silty clay (CL/ML)							
17	Organic silt (OL)							
18	Inorganic silt (MH)							
19	Fat clay (CH)							
20	Organic clay (OH)							
21	Peat (PT)							
22								
23								
24								
25								
26								
27								
28								

Notes: (1) trace <5%; few = 15-25%; little = 15-25%; some 30-45%; mostly 50-100%.
(3) Use a borderline symbol "/" when the percentage of both soil types are estimated to be between 45 and 55%.
(2) Prefix/Suffix can be used when the complete description is referenced. If a prefix/suffix is used the soil classification symbols to enclosed in parenthesis.
Prefix: s=sandy; g=gravelly. Suffixes: s=with sand; g=with gravel; c=with cobbles; b=with boulders. Examples: Sandy lean clay = s(CL);
Poorly graded sand with silt and gravel = (SP-SM)g; poorly graded gravel with sand, cobbles, and boulders = (GP)scb; gravelly silt with sand and cobbles = g(ML)sc

Bay West

Project Name: Sample Graphics
Project Number:
Driller:
Geologist:

Rock Core No.:
Well No.:
Total Depth:
Drilling Method:
Sampling Method:
Grade Elevation:
Date Started/Completed:

Depth	Description - Remarks	Graphic Section	Munsell Color	Recovery/ loss (in.)	Headspace PID/FID/ TLV
	Limestone				
1	Sandstone				
2	Siltstone				
3	Conglomerate				
4	Shale or Mudstone				
5	Basalt				
6	Granite				
7	Gneiss				
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
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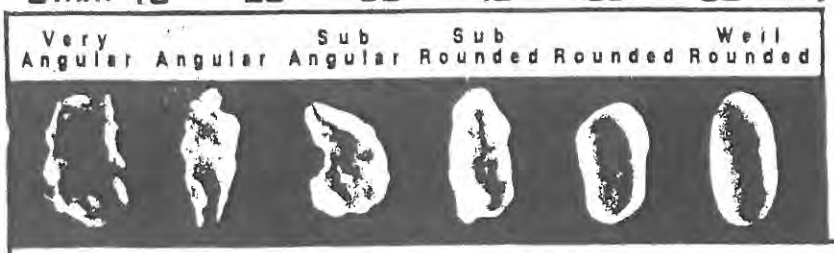
25					
26					
27					
28					
29					
30					

ATTACHMENT C

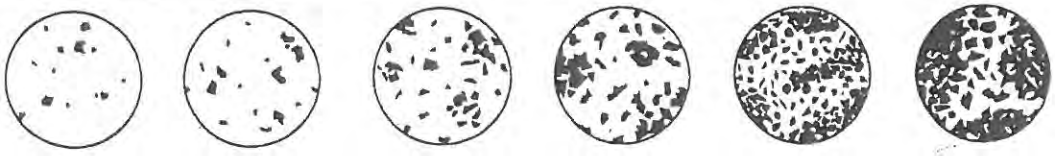
Geotechnical Gauge

0mm 10 20 30 40 50 60 70 80 90 100 110



Geotechnical Gauge

Manuf. by:
W.F. McCollough
3101 Elkridge Ct.
Beltsville, MD 20705



MAJOR DIVISIONS			GRAPHIC SYMBOL	LETTER SYMBOL	TYPICAL DESCRIPTIONS
COARSE GRAINED SOILS (MORE THAN 50% OF MATERIAL IS LARGER THAN NO. 200 SIEVE SIZE)	GRAVEL AND GRAVELLY SOILS (MORE THAN 50% OF COARSE FRACTION RETAINED ON NO. 4 SIEVE)	CLEAN GRAVELS (LITTLE OR NO FINES)		GW	WELL - GRADED GRAVELS, GRAVEL - SAND MIXTURES, LITTLE OR NO FINES.
				GP	POORLY - GRADED GRAVELS, GRAVEL - SAND MIXTURES, LITTLE OR NO FINES.
		GRAVELS WITH FINES (APPRECIABLE AMOUNT OF FINES)		GM	SILTY GRAVELS, GRAVEL - SAND - SILT MIXTURES.
				GC	CLAYEY GRAVELS, GRAVEL - SAND - CLAY MIXTURES.
	SAND AND SANDY SOILS (MORE THAN 50% OF COARSE FRACTION PASSING NO. 4 SIEVE)	CLEAN SAND (LITTLE OR NO FINES)		SW	WELL - GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES.
				SP	POORLY - GRADED SANDS, GRAVELLY SANDS, LITTLE OR NO FINES.
		SANDS WITH FINES (APPRECIABLE AMOUNT OF FINES)		SM	SILTY - SANDS, SAND - SILT MIXTURES.
				SC	CLAYEY SANDS, SAND - CLAY MIXTURES.
FINE GRAINED SOILS (MORE THAN 50% OF MATERIAL IS SMALLERTHAN NO. 200 SIEVE SIZE)	SILTS AND CLAYS	LIQUID LIMIT LESS THAN 50		ML	INORGANIC SILTS AND VERY FINE SANDS, ROCK FLOUR, SILTY OR CLAYEY FINE SANDS OR CLAYEY SILTS WITH SLIGHT PLASTICITY.
				CL	INORGANIC CLAYS OF LOW TO MEDIUM PLASTICITY, GRAVELLY CLAYS, SANDY CLAYS, SILTY CLAYS, LEAN CLAYS.
				OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY.
	SILTS AND CLAYS	LIQUID LIMIT GREATER THAN 50		MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS FINE SAND OR SILTY SOILS.
				CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS.
				OH	ORGANIC CLAYS OF MEDIUM TO HIGH PLASTICITY, ORGANIC SILTS.
HIGHLY ORGANIC SOILS				PT	PEAT, HUMUS, SWAMP SOILS WITH HIGH ORGANIC CONTENTS.

Light Brown

Yellowish Orange

Greenish Gray

Olive Gray

Light Gray

Dark Gray

CLAY

CLAY CONSISTENCY	THUMB PENETRATION	SPT, N BLOWS/FT.	Undrained Shear Strength c_u (PSF) TORVANE	Unconfined Compressive Strength q_u Pocket Penetrometer
VERY SOFT	Easily penetrated several inches by thumb. Exudes between thumb and finger's when squeezed in hand.	< 2	250	500
SOFT	Easily penetrated one inch by thumb. Molded by light finger pressure.	2 - 4	250 - 500	500 - 1000
MEDIUM STIFF	Can be penetrated over 1/4 " by thumb with moderate effort. Molded by strong finger pressure.	4 - 8	500 - 1000	1000 - 2000
STIFF	Indented about 1/4" by thumb but penetrated only with great effort.	8 - 15	1000 - 2000	2000 - 4000
VERY STIFF	Readily indented by thumbnail.	15 - 30	2000 - 4000	4000 - 8000
HARD	Indented with difficulty by thumbnail.	> 30	> 4000	> 8000

SAND

SOILTYPE	SPT, N Blows/ft.	Relative Density,%	FIELD TEST
VERY LOOSE SAND	4	0 - 15	Easily penetrated with 1/2" reinforcing rod pushed by hand.
LOOSE SAND	4 - 10	15 - 35	Easily penetrated with 1/2" reinforcing rod pushed by hand.
MEDIUM DENSE SAND	10 - 30	35 - 65	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.
DENSE SAND	30 - 50	65 - 85	Penetrated a foot with 1/2" reinforcing rod driven with 5-lb hammer.
VERY DENSE SAND	50	85 - 100	Penetrated only a few inches with 1/2" reinforcing rod driven with 5-lb hammer.

Unified Soil Classification System (USCS)

	MILLIMETERS	INCHES	SIEVE SIZES
BOULDERS	> 300	> 11.8	-
COBBLES	75 - 300	2.9 - 11.8	-
GRAVEL:			
COARSE	75 - 19	2.9 - .75	-
FINE	19 - 4.8	.75 - .19	3/4" - No. 4
SAND:			
COARSE	4.8 - 2.0	.19 - .08	No. 4 - No. 10
MEDIUM	2.0 - .43	.08 - .02	No. 10 - No. 40
FINE	.43 - .08	.02 - .003	No. 40 - No. 200
FINES:			
SILTS	< .08	< .003	< No. 200
CLAYS	< .08	< .003	< No. 200

ATTACHMENT D

ASTM Designation: D2488-93

Designation: D 2488 – 93

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (See Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.5 The values stated in inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions:*

3.1.1 Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:

Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and

Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1.2 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.1.3 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.1.4 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.5 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.1.6 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.1.7 *sand*—particles of rock that will pass a No. 4

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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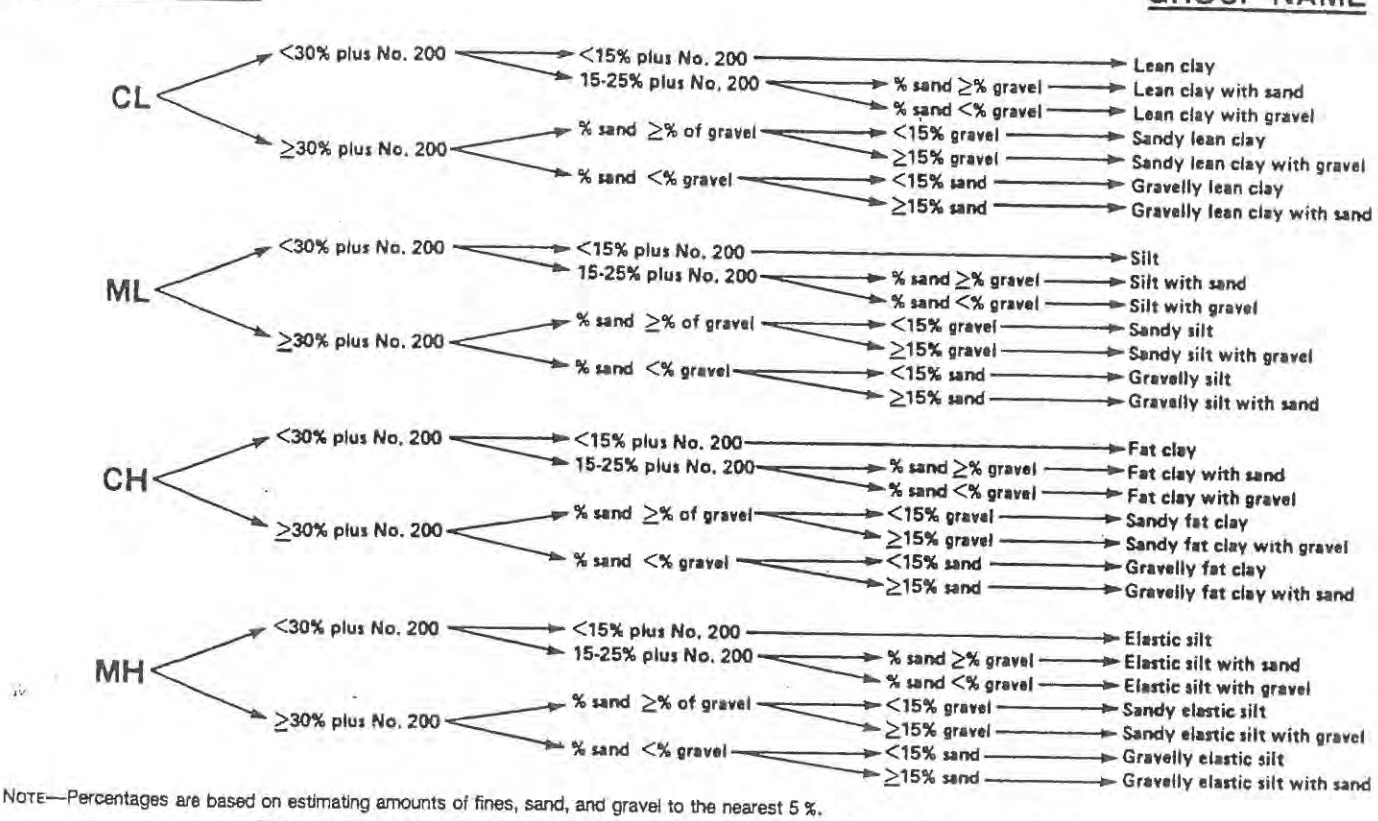
[2] *Annual Book of ASTM Standards*, Vol 04.08.



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)

(4.75-mm) sieve and be retained on a No. 200 (75-μm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-μm) sieve.

fine—passes a No. 40 (425-μm) sieve and is retained on a No. 200 (75-μm) sieve.

3.1.1.8 *silt*—soil passing a No. 200 (75-μm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

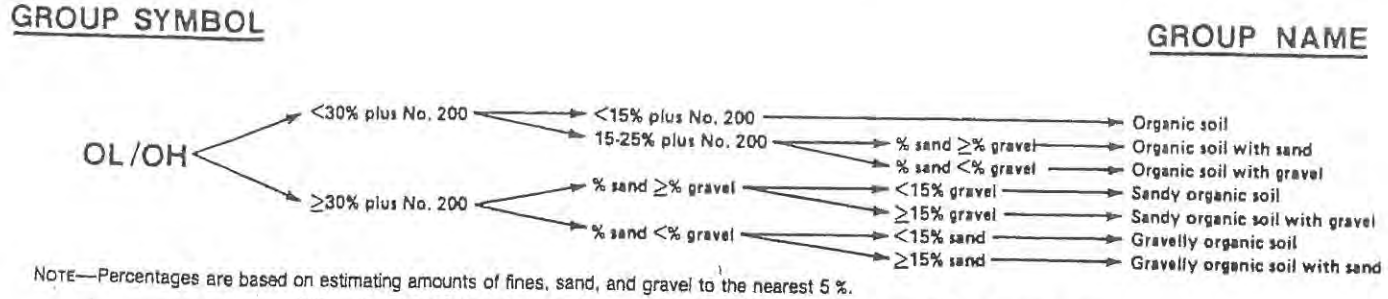
4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Figs. 1a and 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

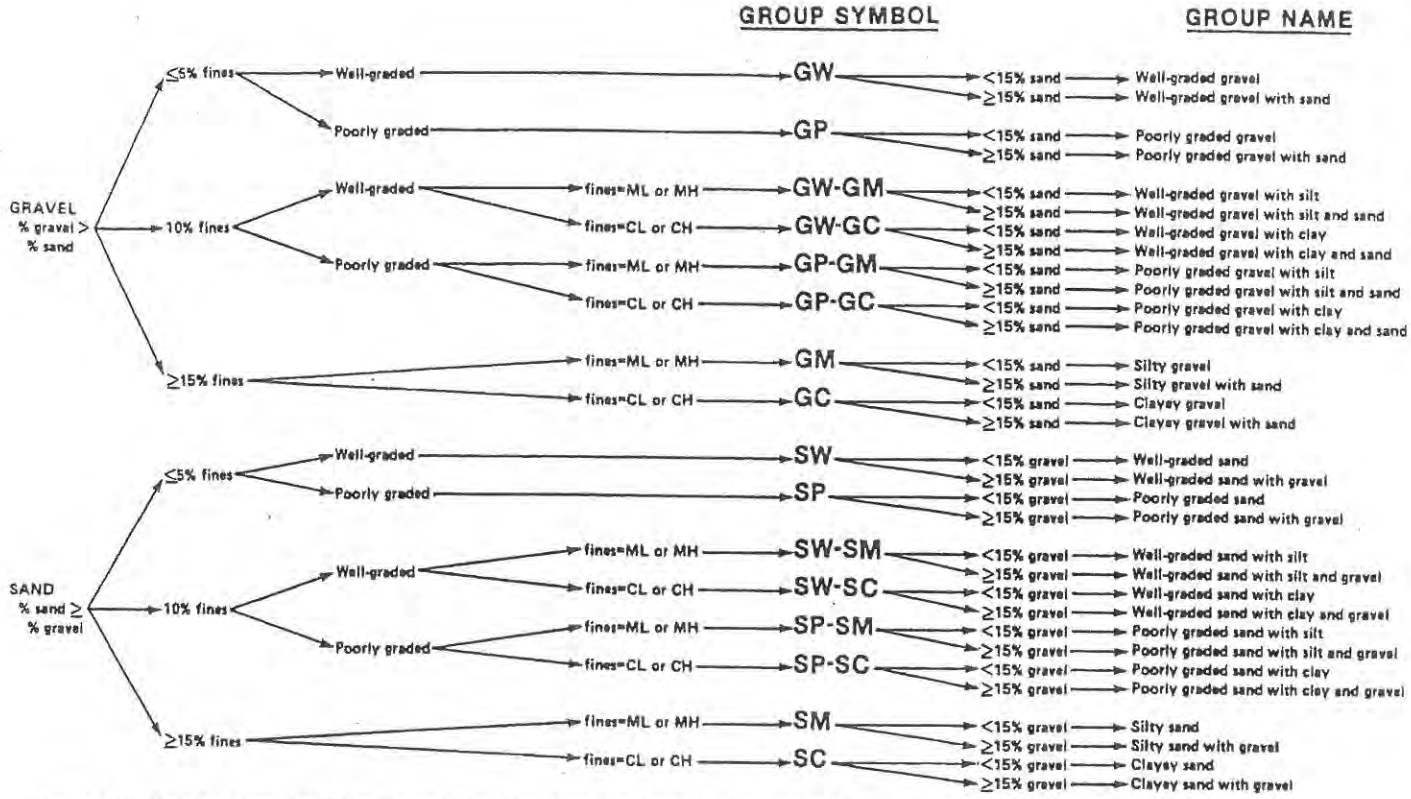
NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)



NOTE—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

6. Apparatus

6.1 *Required Apparatus:*

6.1.1 *Pocket Knife or Small Spatula.*

6.2 *Useful Auxiliary Apparatus:*

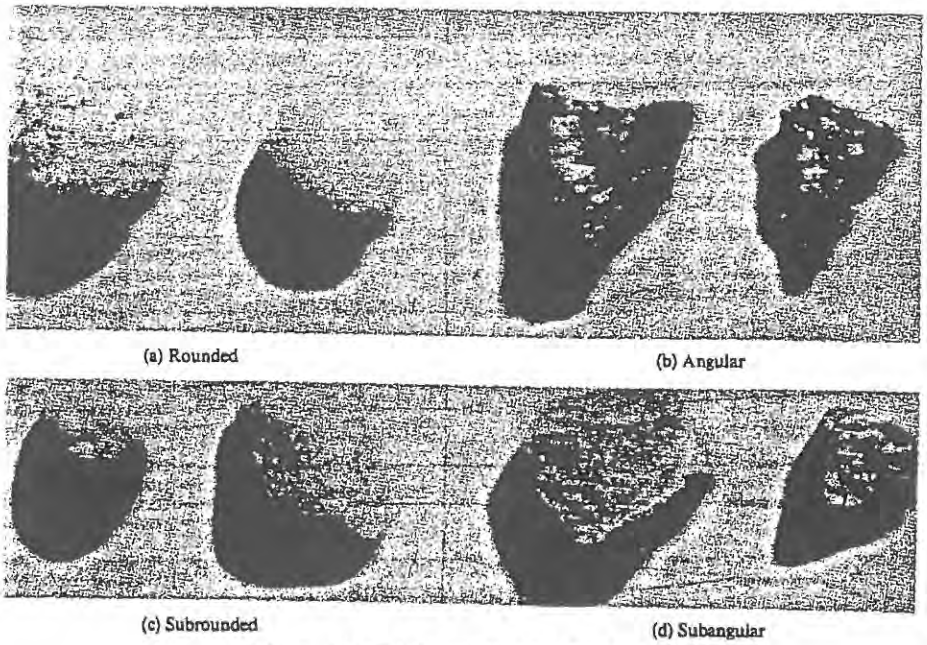
6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.



(a) Rounded (b) Angular

(c) Subrounded (d) Subangular

FIG. 3 Typical Angularity of Bulky Grains

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 Caution—Do not add water to acid.

9. Sampling

9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 5—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 6—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 7—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

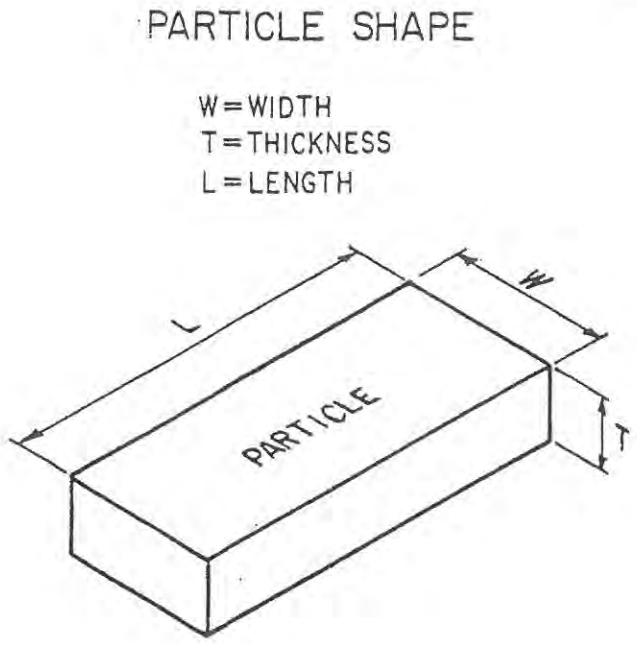


FIG. 4 Criteria for Particle Shape

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼ in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpre-

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Varified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

tation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 8—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 9—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be sidered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength:*

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 10—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy:*

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

TABLE 10 Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness

the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness:*

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils:*

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 11—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils:*

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 12—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Figs. 1a and 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

15. Procedure for Identifying Coarse-Grained Soils (Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

Soil Symbol	Dry Strength	Dilatancy	Toughness
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Medium to high	None to slow	Medium
MH	Low to medium	None to slow	Low to medium
CH	High to very high	None	High

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words "with clay" or "with silt" to indicate the plasticity characteristics of the fines. For example: "well-graded gravel with clay, GW-GC" or "poorly graded sand with silt, SP-SM" (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "poorly graded gravel with sand, GP" or "clayey sand with gravel, SC" (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words "with cobbles" or "with cobbles and boulders" shall be added to the group name. For example: "silty gravel with cobbles, GM."

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 13—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

NOTE 14—Other examples of soil descriptions and identification are given in Appendixes X1 and X2.

NOTE 15—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %

Few—5 to 10 %

Little—15 to 25 %

Some—30 to 45 %

Mostly—50 to 100 %

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size; coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the

percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

X6. RATIONALE

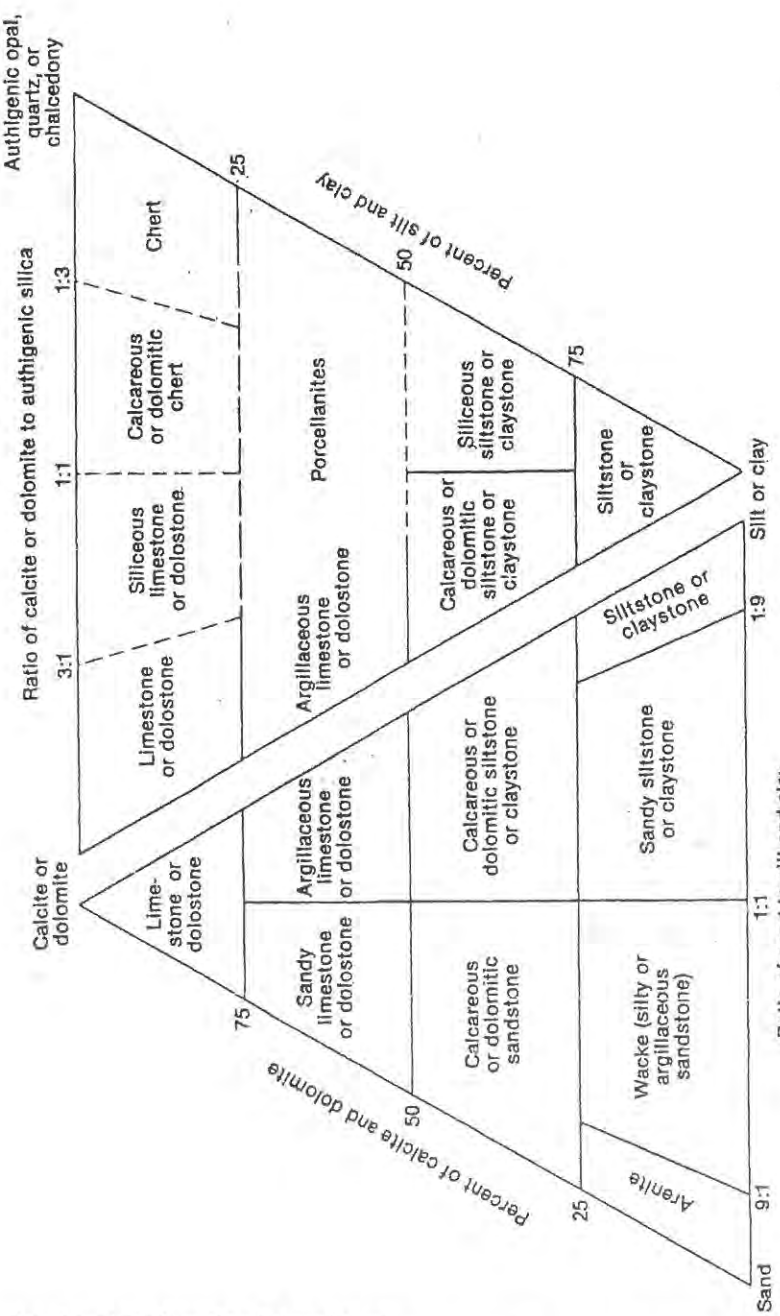
Changes in this version from the previous version, D 2488 – 90, include the addition of X5 on Abbreviated Soil Classification Symbols.

ATTACHMENT E

First Page of the AGI Data Sheets on Sedimentary, Igneous, and Metamorphic Rocks

Names for Sedimentary Rocks

Condensed by R.R. Compton from *Manual of Field Geology*, John Wiley and Sons, New York, 1956



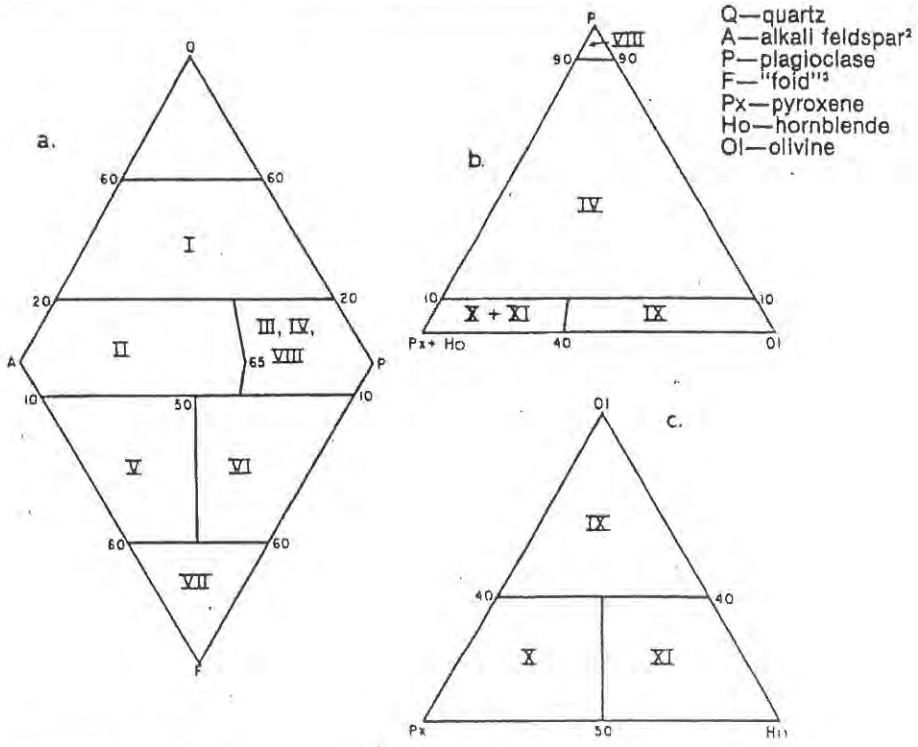
Rock Names for Mixtures of Sedimentary Materials. The sand, silt, and clay do not include detrital grains of calcite or dolomite. Mainly after C.M. Gilbert (*in* Williams, Turner, and Gilbert, 1954, *Petrography:* San Francisco, W.H. Freeman and Co., p. 270.)

Arenite: relatively well sorted sandstone.
Wacke: sandstone so poorly sorted as to include more than 20 percent of silt or clay.
Graywacke: strongly indurated dark-colored wacke.
Shale: siltstone (silty shale) or claystone (clay shale) with prominent bedding cleavage (fissility).
Mudstone: mixture of silt and clay with blocky or spheroidal fracture.
Argillite: highly indurated (generally recrystallized) claystones or siltstones that break into hard, angular fragments.

Field Classification For Igneous Rocks

IUGS Preliminary classification for plutonic rocks[1]

THE PHANERITES[1]



a. General classification.
b. Ultramafic and gabbroic rocks, and anorthosites.
c. Ultramafic rocks.
I, granitoids; II, syenitoids; III, dioritoids; IV, gabbroids; V, foid syenitoids; VI, foid dioritoids and gabbroids; VII, foidolites; VIII, anorthosites; IX, peridotites; X, pyroxenites; XI, hornblendites; II—IV, qualifier "foid-bearing" if foids are present; IX—XI, ultramafic rocks.

In order to plot a rock's composition in the appropriate triangle on "a", the three components alkali feldspar (A), plagioclase feldspar (P), and quartz (Q) *or* the foid minerals (F) are equated to 100 percent—*i.e.*, the other components are subtracted from the total mode and the remaining QAP or FAP percentages are normalized to 100 percent. . . *etc.* (for "b" and "c").

Diagrams for the general nomenclature are presented on Data Sheet 48. Additional diagrams outlining suggested use of prefixes leuco- and mela- and giving nomenclature for less common phanerites such as carbonatites and lamprophyres may be found in the following references:

Dietrich, R.V. and Skinner, B.J., 1979, Rocks and Rock Minerals: Wiley, N.Y., 369p.

IUGS Subcommission on the Systematics of Igneous Rocks, 1973, Classification and nomenclature of plutonic rocks: Geotimes, v. 18, n. 10 (Oct.), p. 26-30.

Streckeisen, A., 1976, To each plutonic rock its proper name: Earth Science Rev., v. 12, p. 1-33.

1. In the IUGS scheme, "plutonic rock", which refers to phaneritic rocks, is not assumed to require an igneous origin.

2. Alkali feldspar includes potassium feldspars, perthite (including its plagioclase component), and anorthoclase.

3. "foid" includes the feldspathoids—leucite and pseudoleucite, nepheline, sodalite, nosean, hauyne, cancrinite, analcime, *etc.*

Descriptive Classification of Metamorphic Rocks*

by Robert R. Compton, Stanford University

In this binomial system for naming metamorphic rocks, the main rock name is based on the texture of the rock, and the principal or more significant minerals are added as modifying nouns, as in biotite-quartz schist or andalusite-cordierite hornfels. The names are meant to be applied on a descriptive basis; a schistose rock, for example, should not be called a hornfels just because it is found in a contact aureole.

TEXTURES

Schistose—grains platy or elongate and oriented parallel or subparallel. *Foliated* (lepidoblastic) if fabric is planar, *lineated* (nematoblastic) if linear.

Granoblastic—grains approximately equidimensional; platy and linear grains oriented randomly or so subordinate that foliation is not developed.

Hornfelsic—grains irregular and interincluded but generally microscopic; recognized in field by unusual toughness, ring to hammer blow, and hackly fracture at all angles. Under hand lens, freshly broken surfaces show a sugary coating that will not rub off (formed by rending of interlocking grains).

Semischistose (gneissic)—platy or linear grains subparallel but so subordinate or so unevenly distributed that rock has only a crude foliation; especially common in metamorphosed granular rocks, such as sandstones and igneous rocks.

Cataclastic—clastic textures resulting from breaking and grinding with little if any recrystallization; characterized by angular, lensoid, or rounded fragments (*porphyroclasts*) in a fine-grained and commonly streaked or layered groundmass. *Mortar structure* applies to nonoriented arrangements, and *phacoidal, flaser,* and *augen structure* apply to lenticular arrangements.

ROCK NAMES

SCHISTOSE ROCKS

Schist—grains can be seen without using a microscope.

Phyllite—all (or almost all) grains of groundmass are microscopic, but cleavage surfaces have sheen caused by reflections from platy or linear minerals; commonly corrugated.

Slate—grains are microscopic; very cleavable; surfaces dull; tougher than shale and cleavage commonly oblique to bedding.

Phyllonite—appearance like phyllite but formed by cataclasis (see mylonite) and recrystallization commonly of coarser-grained rocks, as indicated by relict rock slices, slip folds, and porphyroclasts.

GRANOBLASTIC ROCKS

Granulite or **granofels**—granoblastic rocks, irrespective of mineral composition; because *granulite* can connote special compositions and conditions of origin, *granofels* may be preferred.

Quartzite, marble, and amphibolite—compositional names that generally connote granoblastic texture; exceptions should be modified for clarity, as *schistose quartzite* or *plagioclase hornblende schist.*

Tactite (skarn)—heterogeneous calc-silicate granulites and related metasomatic rocks of typically uneven grain.

HORNFELSIC ROCKS

All called *hornfels*, or, if relict features are clear, hornfelsic may be used with the original rock name (as *hornfelsic andesite*).

SEMISCHISTOSE (GNEISSIC) ROCKS

Semischist—fine-grained (typically less than 1/4 mm) so that individual platy or lineate grains are indistinct; relict features often common.

Gneiss—generally coarser than 1/2 mm with small aggregates of platy or lineate grains forming separate lenses, blades, or streaks in otherwise granoblastic rock. Platy or lineate structures may be distributed evenly through the rock or may be concentrated locally so that some layers or lenses are granoblastic or schistose (*banded gneiss*).

*Modified after Data Sheet 27,1965, which was condensed from *Manual of field geology,* John Wiley & Sons, New York, 1962

CATACLASTIC ROCKS

Where original nature of rock is still apparent, rock name can be modified by suitable adjectives (as *cataclastic granite, flaser gabbro, phacoidal rhyolite*).

Mylonite—crushing so thorough that rock is largely aphanitic and commonly dark-colored; may be layered and crudely foliated but not schistose like phyllonite; porphyroclasts commonly rounded or lenticular.

Ultramylonite, pseudotachylyte—Aphanitic to nearly vitreous-appearing dark rock commonly injected as dikes into adjoining rocks.

RELICT AND SPECIAL TEXTURES AND STRUCTURES

If textures of low-grade metamorphic rocks are dominantly relict, original rock names may be modified (as *massive metabasalt, semischistose meta-andesite*). If hydrothermal alteration has produced prominent new minerals, names such as *chloritized diorite* and *sericitized granite* can be used.

Strongly metasomatized rocks with coarse or unusual textures may require special names such as *greisen, quartz-schorl rock*, and *corundum-mica rock*.

Migmatite—a composite rock composed of igneous or igneous-appearing and/or metamorphic materials that are generally distinguishable megascopically.

ATTACHMENT F

Example of Completed Soil Boring Log and Completed Coring Log

Bay West

Project Name: Zurn Lake
Project Number: J8675309
Driller: Jeremy Brandt-NTS
Geologist: Scott Zurn

Soil Boring No.: GP-6
Well No.:
Total Depth: 24 ft bgs
Drilling Method: Geoprobe
Sampling Method: 4 ft Macro Core
Grade Elevation:
Date Started/Completed: 1610 8-19-02 / 1815 8-19-02

Depth	Description - Remarks	Graphic Section	Munsell Color	USCS Class.	Analytical Sample	REC (in.)	Blows	Headspace PID/FID/ TLV
	Sandy silt s(ML), fine to medium grained sand, black, trace fine gravel, dry. Grass surface, topsoil.		5YR2.5/1	s(ML)		24	2	0
1	Sandy silt s(ML), fine to medium grained sand, reddish brown, trace fine gravel, moist.		5YR5/3				3	
2	No Recovery.					NR	8	NR
3								
4	Sandy silt s(ML), fine to medium grained sand, reddish brown, some fine to coarse gravel, moist		5YR5/3		GP-6 4'-6'	24	5	0
W.L. 5	to wet, no odor.							
6	Same As Above (SAA).		5YR5/3			8	8	0
7								
8	SAA.		5YR5/3			24	10	0
9								
10	SAA.		5YR5/3			14	9	0
11								
12	Silty sand and gravel (SM/GW), fine to medium grained sand and fine to coarse angular gravel,		7.5YR5/3	SM/GW		24	25	0
13	brown, wet, no odor.							
14	No Recovery (pushed a rock)					NR	40	NR
15								
16	Sandy silt (ML), fine grained sand, reddish brown, few gravel, wet.		5YR5/3	ML		24	20	0
17	Poorly graded sand (SP), medium grained sand, rounded, dark brown, wet, no odor.		7.5YR3/4	SP				
18	Poorly graded sand (SP), very fine to fine grained sand, rounded to sub rounded, brown, wet.		7.5YR5/3			14	22	0
19								
20	SAA		7.5YR5/3			24	20	0
21								
22	Well graded sand (SW) fine to coarse grained sand, rounded, dark brown, wet, no odor.		7.5YR3/4	SP	GP-6 22'-24'	18	30	0
23								
24	-End of boring at 24 ft bgs due to refusal. Set temporary PVC well screen at 0' to 11.5' bgs. Initial							
25	water level measurement was 5.5 ft bgs. Collected grain size analysis from 8-12 ft bgs.							
26								
27								
28								
29								
30								

Bay West

Project Name: Zurn Lake
Project Number: J8675309
Driller: Rocky Balboa
Geologist: Scott Zurn

Rock Core No.: C1
Well No.:
Total Depth: 14
Drilling Method: Core
Sampling Method: C
Grade Elevation: 800 feet ASL
Date Started/Completed: 0800 11-14-02 / 1600 11-15-02

Depth	Description - Remarks	Graphic Section	Munsell Color	Recovery/ loss (in.)	Headspace PID/FID/ TLV
	See Soil Boring Log B1 for Soil Classification				
1					
2					
3					
4					
5					
6					
7 WL					
8	Fossiliferous Limestone, Platteville Formation light brown, wet.		7.5YR6/3	8	45
9	Shale, Glenwood Formation weathered, olive brown, moist.		2.5Y4/3	10	0
10	Shale, Glenwood Formation olive, moist.		5Y5/4	12	0
11	SAA		5Y5/4	12	0
12	SAA		5Y5/4	12	1
13	Sandstone, St. Peter, fine to medium grained, light brown, rounded and subrounded grains, poorly cemented, wet.		7.5YR6/3	12	0
14	-End of boring at 14 ft bgs. Water level measurement 7.5 ft bgs.				
15					
16					
17					
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27					
28					
29					
30					

Bay West

Standard Operating Procedure

Sediment Sampling

CORP-ENV-019-1543704

Revised: June 14, 2012

Review and Approval:

Developed by: _____
Signature
QA/QC Manager
Title
Date: June 14, 2012

Reviewed by: _____
Signature
QA/QC Manager
Title
Date: June 14, 2012

Approved by: _____
Signature
Vice President of Operations
Title
Date: June 14, 2012

Questions and requests for information regarding this Standard Operating Procedure (SOP) should be directed to the Vice President of Operations or the QA/QC Manager. This document cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

This Standard Operating Procedure (SOP) provides a standardized method for collecting sediment samples at suspected hazardous waste sites. This SOP must be used by samplers trained in sample collection, sample handling and sample custody. Deviations from the procedures outlined in this document must be approved by the Field Supervisor and/or the Project Manager prior to initiation of the sampling activity.

This SOP is applicable to the collection of representative sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- toxicity;
- biological availability and effects of contaminants;
- benthic biota;
- extent and magnitude of contamination;
- contaminant migration pathway and potential source;
- fate of contaminants; and
- grain size distribution.

1.2 Scope

The methodologies discussed in this SOP are applicable to the sampling of sediment in both flowing and standing water. For the purposes of this procedure, sediments are mineral and organic particulate matter transported by fluid flows which are eventually deposited as a layer of solid particles on the bed or bottom of a body of water. The water may be static, as in lakes, ponds, and impoundments; or flowing, as in rivers and streams. The Bay West, Inc. SOP, *Water Hazards*, CORP-H&S-019-62590 should be reviewed prior to sampling (Bay West, 2003).

2.0 DEFINITIONS

Sediment – particulate matter transported by fluid flow which is eventually deposited as a layer of solid particles on the bed or bottom of a body of water.

Dredge – a mechanical device used to bring sediment to the surface.

Coring Device – a tubular device used to collect an undisturbed column of sediment.

3.0 RESPONSIBILITIES

3.1 Sampler

The sampler is responsible for the collection of representative samples, correct sample labeling, and the recording of the sampling data as specified in this SOP.

3.2 Field Supervisor

The Field Supervisor is responsible for overseeing the sediment sampling activities. The Field Supervisor responsible for checking all work performed by the sampling team and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Work Plan, QAPP and/or the Field Sampling Plan. It is the responsibility of the Field Supervisor to communicate with the sampling team regarding specific collection objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the Field Supervisor to communicate the need for any deviations from the Project Plan with the Project Manager. The Field Supervisor will ensure that every team member will have had the appropriate training to execute the sampling event and all sampling activities are performed safely in accordance with the site-specific SSHP.

3.3 Project Manager

The Project Manager is responsible for the overall success of the field program and will ensure the maintenance of logbooks and forms and assessing and approving techniques not specifically described in this SOP. Direct contact with the client will be administered by the Project Manager.

4.0 REQUIRED EQUIPMENT

This section provides a list of commonly used equipment for sediment sampling. It is not comprehensive as additional equipment may be necessary depending on site conditions or the client's needs.

4.1 Sampling Devices

- Ponar® dredge – used for collecting sediment samples in hard bottoms such as sand, gravel, and clay. Designed for severe environmental conditions, including salt water use.
- Ekman® bottom sampler – used for collecting sediment samples in soft, finely divided bottoms. Does not work well in bottoms that are vegetated or have coarse debris. An extension handle is available to aid sampling in shallow water.
- Sediment coring sampler – this type of sampler has a removable inner sleeve made of Teflon® or acetate. Remove sediment core intact for geological testing and environmental sub-sampling.
- VOC sampling coring device – used for collecting sediment samples designated for volatile organic compounds (VOCs) analysis or toxicity testing. An EnCore® or a TerraCore® sampler may be used, or a liner sleeve may be sent to the lab for subsampling.

- Nylon rope or steel cable – for raising and lowering the dredge.
- Sample collection containers – Laboratory supplied 4-oz., 8-oz., and/or one-litre wide mouth amber glass jars with Teflon lined lids. Samples designated for VOC analysis or toxicity testing must be submitted in a sealable sampler, a pre-preserved vial or the Teflon® sleeve used for collection.
- Gloves – for personal protection and to prevent cross-contamination of samples. Disposable powderless gloves as specified in the SSHP should be used for sampling.
- Field clothing and personal protective equipment – as specified in the SSHP. Personal floatation devices (PFDs) must be worn for all work performed from a boat.
- Sampling flags – Used for identifying sediment sampling locations in very shallow water. Buoys with anchors may be required in deeper water.
- Field notebook – a bound book used to record progress of sampling effort and record any problems and field observations during sampling.
- Permanent marking pen – used to mark sample bottle labels, sediment boring tubes and for documentation in the of field logbooks.
- Stainless steel spoon – or equivalent. Used for homogenizing sediment samples that will not be used for VOCs analysis or toxicity testing.
- Stainless steel bowls – or equivalent. Used for compositing samples that will not be used for VOCs analysis or toxicity testing. Must have sufficient capacity to contain a full dredge sample.

5.0 PROCEDURES

A new pair of gloves and eye protection are to be worn at each sampling location. Splash protection may be required by the SSHP. Each sampling location must be recorded on the site diagram prior to collecting the sample. All sampling equipment must be decontaminated prior to use.

5.1 Sediment Collection with a Dredge

The dredge samplers consist of spring-loaded semi-cylindrical jaws that close either when they encounter the sediment while being lowered down through the water or are actuated by a messenger weight that is dropped along a line to the sampler. In either case, the jaws close and remove a portion of the sediment ranging in depth up to approximately eight inches with depth being dependent on the physical characteristics of the sediment and the size and weight of the sampler.

Some precautions must be taken when using a dredge-type of sampler. First, once the jaws of the sampler have been spread and set, care must be taken when handling the device not to accidentally trip the triggering device, which could result in injury. Secondly, the quality of the samples is largely dependent on the speed at which the sampler is lowered to the sediment and the speed at which it is withdrawn from the sediment. Allowing the sampler to fall to the sediment quickly can result in significant shock waves that can blow soft/loose sediment from the sample location. Withdrawing the sampler back up through the water too quickly can result

in turbulence in the water above the sampler that can wash the sediments out of the sampler. Also, sediments containing coarse gravel, wood, and other debris can limit the sampling effectiveness by preventing complete closure of the sampler jaws.

Attach a sturdy nylon rope or steel cable to the ring provided on top of the dredge. Arrange the dredge with jaws in the open position, setting the tripping device so that the sampler remains open when lifted from the top. If the dredge is so equipped, place the spring loaded pin into the aligned holes in the trip bar. Slowly lower the sampler to a point approximately two inches above the sediment. Drop the sampler to the sediment. With automatic samplers, the jaws of the sampler will close as the line slackens. If the sampler uses a messenger weight, once the line slackens, re-tension the line slightly, and send the messenger down the line to trip the sampler. Some samplers have a spring loaded pin; by pulling up sharply on the line, dredge is closed. Slowly raise the dredge to the surface and slowly decant any free liquid through the screens on top of the dredge. Care should be taken to retain the fine sediment fraction during this operation.

Open the dredge and transfer the sediment to a stainless steel bowl or other compositing container. If necessary, repeat the collection procedure until sufficient material has been collected. Homogenize the sample by mixing with a stainless steel spoon or equivalent, and then transfer the sample to a laboratory supplied glass jar. When splitting sediment samples, continuous mixing may be required to maintain homogeneity, and to avoid the settling of larger sediment fractions in the bottom of the compositing bowl. Affix a sample ID label to each container and copy the information to the Field Data Sheet.

5.2 Collection with a Coring Device

Sediment designated for VOC analysis or toxicity testing must be collected with a sampling system consisting of a tube sampler, removable Teflon® or acetate tube liner, 'eggshell' check valve, nosecone, extensions, and a "T" handle or drive hammer. The use of additional extensions can increase the depth of water from which sediment can be collected from 24 inches to 10 feet or more, but sample handling and manipulation become more difficult as the depth of water increases.
To collect the sample using the tube liner sampler, press down on the sampling device until the desired depth is achieved. After reaching the desired depth, rotate the sampler to shear off the bottom of the sediment core. Slowly withdraw the sampler from the sediment, and decant the surface water, using care to retain the fine sediment fraction. Unscrew the nosecone and remove the eggshell check valve. Slide the liner core out of the sampler tube. If there is headspace in the upper end of the sediment core, use a hacksaw to shear off the tubing at the sediment surface. Cap both ends of the tube. To minimize the potential for volatilization of certain compounds of interest, samples designated for VOC analysis or toxicity testing must not have any space present between the sediment core and the end of the cap on the liner sleeve.

If the drive hammer is used, insert the tapered handle (drive head) through the top of the sampler. Drive the sampler into the sediment to the desired depth, and rotate the sampler to shear off the bottom of the sediment core. Slowly withdraw the sampler from the sediment, and decant the surface water, using care to retain the fine sediment fraction. Unscrew the

nosecone and remove the eggshell check valve. Slide the liner core out of the sampler tube. If there is headspace in the upper end of the sediment core, use a hacksaw to shear off the liner tubing at the sediment surface. Cap both ends of the tube.

Secure the caps on both ends of the core with tape, and use a waterproof pen to indicate the orientation of the sediment core. Affix a sample identification label to the core and copy the information to the Field Data Sheet. Immediately place the sample on ice for transport to the analytical laboratory.

5.3 Collection of VOC samples using a Coring Device and an EnCore® or TerraCore® Sampler

VOC samples are collected from the recovered core by subsampling with an EnCore® or TerraCore® Sampler. If the sediment is sufficiently cohesive, split the core lengthwise with a sharp blade and collect a sample using the EnCore® or TerraCore® Sampler. Follow the manufacturer's instructions for sealing the sampling device or place the sample aliquot into a pre-weighed, pre-preserved vial. Place the sampler or vial in a zipper-top bag and place the sample in a chilled cooler. Non-cohesive sediment samples may need to be transferred directly into pre-weighed vials supplied by the laboratory.

5.4 Decontamination Procedures

The objectives of decontamination are to prevent the introduction of contaminants into samples from sampling equipment or other samples. Disposable sampling equipment will be used whenever possible. Samplers will change sample gloves and discard all disposable sampling equipment after each sample is collected to minimize the potential for media cross-contamination. Disposable sampling equipment includes sample gloves, polyethylene scoops and plastic bags.

Non-disposable sampling equipment such as dredges and coring tubes will be decontaminated in accordance with the Bay West SOP *Field Equipment Decontamination at Non-radioactive Sites* SOP CORP-ENV-002-65422V1.

6.0 DOCUMENTATION

The sampler is responsible for complete documentation of the sampling event. It is critical this information is accurate and the necessary information is communicated to the laboratory.

6.1 Field Logbook

The field logbook will contain all of the information collected during the sampling event. This includes, but is not limited to: arrival and departure times of all people on site, tailgate meetings, equipment checks and calibrations, weather conditions, a record of all measurements taken, sample names and times, deliveries and pickups of equipment/samples, and descriptions of any deviations to the Sampling Plan and the reason for the deviation.

Some projects may require site specific forms to be filled out. As with the logbook, all forms should be filled out legibly with errors crossed out with a single line, initialed and dated. Forms should be kept in a 3-ring binder, if possible, to avoid misplacement or loss.

6.2 Sample Labeling

Correct sample labeling is critical to proper sample identification and accurate log-in at the lab. Sample labels will be provided with the sample bottles from the lab. Be sure you have a sufficient supply. The required information for the sample label will be spelled out in the Work Plan/QAPP.

In general, the sample label will require:

1. Project / Site name;
2. Sample name formatted as required (refer to the SAP);
3. Date and time sampled;
4. Requested analyses;
5. Any preservatives used inside the sample bottle;
6. Sampler's initials;

After the sample label is applied, the bottle should be wrapped with packing tape to minimize the chance of the label separating from the bottle. All samples should be stored on wet ice at 4°C ± 2°C in a secured cooler. Samples designated for VOC analysis or toxicity testing must be kept away from direct sunlight and immediately chilled to 4°C ± 2°C. Ship samples under chain-of-custody, protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

6.3 Chain of Custody

The information on the Chain of Custody should match the bottle label EXACTLY. Double check to see if this is the case. Samples must be kept secure until delivered to the lab or picked up by a courier/overnight shipper. The cooler must be sealed with strapping tape and custody seals applied on opposite corners. Follow procedures in Bay West, Inc. SOP, *Packaging and Shipping of Environmental Samples*, CORP-ENV-006-1510206 (Bay West, 2012).

7.0 PROCEDURE PERFORMANCE EXPECTATIONS

The number and type of quality control samples described in the SAP will be collected at the required frequency.

7.1 Duplicate Samples

Duplicates samples are used to check on sampling and laboratory precision. For analyses other than VOCs, the sediment must be thoroughly mixed before being placed in the sample jars. VOC samples should be collected as close together as possible from the sediment or from the core sampler. These samples are handled with a minimum of disturbance to avoid loss of volatile compounds.

7.2 Matrix Spike Samples

Matrix spike samples measure the ability of the extraction procedures to remove analytes of concern from the matrix in addition to measuring analytical precision. In general, matrix spike samples require triple sample volume for analysis. Mix the sample thoroughly (except for VOCs) prior to filling the three sample jars. Note on the Chain of Custody which sample was selected for a matrix spike.

7.3 Sample Preservation and Handling

Sample preservation procedures are used to maintain the original character of analytes during storage and shipment. Regardless of the nature of the sample, absolute stability for all constituents cannot be achieved. Preservation techniques, such as pH control and refrigeration, may retard physiochemical and biochemical changes. As a general rule, analyzing the sample as soon as possible is the best way to minimize physiochemical and biochemical changes.

All samples will be placed in the appropriate sample container and refrigerated (on ice or ice substitute in a cooler) immediately upon sample collection. All sample containers will be placed into individual re-sealable plastic zip-top bags. The samples will be transferred to the contract laboratory via commercial express delivery service. The contract laboratory will meet all specified holding times and should make every effort to prepare and analyze the samples immediately after they are received. Chemical preservation, sample container type, and temperature requirements for the analyses to be performed are provided in the site-specific Work Plan and/or QAPP.

Samples will remain in the possession of the sampling personnel until they are shipped to the laboratory. Immediately after collection, and during shipment to the analytical laboratory, samples will be stored in coolers on ice. Ice packaged in plastic storage bags will maintain the temperature in the shipping containers at 4°C ± 2°C. A temperature blank and trip blank will be included in each cooler containing samples for volatiles analysis. Ice will be replenished as necessary to ensure adequate cooling of samples during storage and shipping.

8.0 REFERENCES

2003 Bay West SOP, Field Decontamination at Non-Radioactive Sites, CORP-ENV-002-65422V1, January 27, 2003

2003 Bay West SOP, Water Hazards, CORP-H&S-019-62590, April 18, 2003

2012 Bay West SOP, Packaging and Shipping of Environmental Samples, CORP-ENV-006-1510206, February 10, 2012

Bay West

Standard Operating Procedure

Investigation-Derived Waste

CORP-ENV-018-129394

Revised: October 2, 2012

Review and Approval:

Developed by: _____
Signature
QA/QC Manager
Title
Date: October 2, 2012

Reviewed by: _____
Signature
Project Manager
Title
Date: October 2, 2012

Approved by: _____
Signature
Vice President of Operations
Title
Date: October 2, 2012

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

NOTE: This SOP is current as of the date printed on the bottom. Bay West personnel may produce paper copies of this procedure printed from the controlled document electronic file located on the Intranet. However, it is their responsibility to ensure that they are trained and utilize the current version of this procedure.

TABLE OF CONTENTS

Attachments:

Uniform Hazardous Waste Manifest
Non-Hazardous Waste Manifest
Straight Bill of Lading

1.0 INTRODUCTION

1.1 Purpose

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures employed by Bay West, Inc. to manage investigation-derived waste (IDW) in accordance with state and federal regulations, including the U. S. Environmental Protection Agency (EPA), and Resource Conservation and Recovery Act (RCRA).

1.2 Scope

The scope of this SOP includes the management of all IDW generated by Bay West, Inc.

2.0 DEFINITIONS

IDW includes all materials generated during performance of an investigation or corrective action that cannot be effectively re-used, recycled, or decontaminated in the field. IDW consists of materials that could potentially pose a risk to human health and the environment (e.g., sampling and decontamination wastes), as well as materials that have little potential to pose risk to human health and the environment (e.g., sanitary solid wastes).

Two types of IDW are generated during field activities: indigenous and non indigenous.

- Indigenous IDW expected to be generated during the investigations and remedial activities and include drill cuttings, residual soil samples, soil from trenching and excavations, residual sediment samples, and groundwater from monitoring well development and purging.
- Non-indigenous IDW consists of decontamination rinse fluids; polyvinyl chloride pipe, concrete, and metal from monitoring well plugging and abandonment; and compactable and miscellaneous trash, including personal protective equipment (PPE) (gloves, Tyvek, paper towels, etc.).

Procedures to be utilized for managing IDW are described below.

3.0 RESPONSIBILITIES

All wastes generated during environmental investigations will be managed in accordance with all federal, state, and local generator requirements.

4.0 PROCEDURES

All indigenous solid IDW (soil, rock cuttings, and waste material) generated from borehole drilling activities or excavations will be segregated by location and by visual determination of degree of potential contamination. All indigenous solid IDW will be contained in properly identified U. S. Department of Transportation (DOT) approved open-top steel 55-gal drums and sealed with steel ring bolt-through lids, or lined, steel rolloff boxes equipped with a tarp or other cover device, staged temporarily on and under plastic sheeting (minimum 6-mil thickness) at the site until work is completed, as specified in the site-specific Work Plan (WP).

The temporary stockpiling of soil will be in a manner that is protective of human health and the environment and compliant with stormwater best management practices.

Potentially hazardous soil IDW from drilling or excavation will be identified based on:

(1) process knowledge,
(2) field visual inspection of the soil and waste materials (i.e., heavy discoloration, oil saturated, etc.),
(3) the types of waste materials unearthed (i.e., drum containers, paint or aerosol cans, munitions wastes, etc.), and
(4) screening using field screening instruments (e.g., organic vapor analyzer).

Non-hazardous soil IDW with contaminant concentrations below regulatory criteria may be subject to beneficial re-use and returned to the excavation or used as cover.

All liquid indigenous (groundwater) IDW generated from monitoring well installation, development, and purging will be segregated by sample station. All liquid indigenous IDW will be collected in either properly identified DOT-approved, 55-gal closed-top drums or in properly identified polyethylene storage tanks.

All solid non-indigenous (expendable sampling equipment and trash) IDW will be segregated as non contaminated and potentially contaminated material. Potentially contaminated and non-contaminated solid non-indigenous IDW will be identified in the field on the basis of visual inspection (e.g., soiled versus non-soiled), usage of the waste material (e.g., outer sampling gloves versus glove liners), and field screening of the material using available field instrumentation (e.g., organic vapor analyzer).

All solid non-indigenous, non-contaminated IDW will be segregated from potentially contaminated non-indigenous IDW. Depending on the volumes generated, solid non-indigenous IDW will be contained in properly identified, DOT-approved, steel open-top 55 gal drums and sealed with steel ring bolt-through; lined, steel rolloff boxes with tarp of other cover device; or placed on pallets (pipe, steel equipment, etc.) and covered with plastic.

All liquid non-indigenous (decontamination rinse water) IDW will be segregated by waste stream (e.g., soap and water/water rinses from methanol and hydrochloric acid rinses) and contained in either properly identified proper DOT-approved, 55-gal closed-top drums or in polyethylene storage tanks. All known potentially hazardous liquid non-indigenous IDW streams, such as methanol, hydrochloric acid rinses, and acetone waste from field laboratories, will be contained separately in properly identified proper DOT-approved, closed top 55-gal drums or proper DOT-approved, 5-gallon containers.

The anticipated IDW that will be generated from a field activity and the specific method(s) used to containerize IDW streams during each investigation will be identified in the site-specific WP based on the waste containment options defined above.

5.0 IDW CONTAINER IDENTIFICATION

Each IDW drum container will be marked on the top with an indelible paint marker with the words "Pending evaluation" and the following information:

(1) Project Name
(2) Project Number
(3) Contents
(4) Unique ID Number
(5) Generation Date
(6) Other unique information as required per the Work Plan (i.e., source of waste, source location, sample number, PM, PM phone number)

An inventory of each container containing the above information will be required as part of the field documentation. In the event that a bulk container (rolloff or tote) is utilized the above information will be required to be on paper and placed in a weather resistant zip-lock bag and affixed to the bulk container using a weather resistant tape.

6.0 IDW STORAGE

As per the Work Plan, a designated, agreed upon staging area will be used to store the IDW containers until proper disposal is arranged and ultimate shipment of the containers off-site. A designated Bay West staff person will inspect and document the condition of the staging area on a daily basis and inspect each IDW container for defects including: deterioration, damage, bulging, cracking or any other potential container failure that may result in a leaking container.

7.0 WASTE CHARACTERIZATION, DISPOSAL AND TRANSPORTATION

Until it is identified or characterized, a material is considered extremely hazardous. Liquids must be considered hazardously volatile until shown otherwise. All materials must be considered corrosive to the skin and toxic upon contact until proven otherwise. Until shown otherwise, unknown materials may be water-reactive, pyrophoric, hypergolic, radioactive, shock-sensitive, polymerize spontaneously, or exhibit other exothermic reactions. Changing conditions of temperature, pressure, humidity, air, or light can trigger instability.

Once the material has been characterized it will be properly profiled for disposal to an approved disposal end-facility. Waste profiling will be performed by staff with expertise in waste management procedures and reviewed by Bay West waste disposal coordinators. Bay West waste disposal coordinators will have completed OSHA 29 CFR 1910.120 training, DOT 49 CFR 172 training and have a minimum of 5 years of experience managing hazardous wastes. Proper disposal method(s) for IDW will be based on the type of material and in collaboration with the generator.

Mandatory review of the shipping documents by Bay West waste disposal coordinators is required for all shipments. Proper shipping documents will be prepared onsite prior to review and review will occur prior to transportation. Bay West staff with expertise in waste management

procedures will perform the transport of the containers. Shipping documents will include manifests, labels, markings, land disposal restriction forms and/or any form of lading. Waste profiles and shipping documents will be required to be reviewed and signed by a designated representative of the generator of the waste.

All waste will be properly managed according to local, state, and federal EPA and DOT regulations.

8.0 REFERENCES

Title 49, Code of Federal Regulations, Subtitle B, Chapter 1 – Pipeline and Hazardous Materials Safety Administration, Department of Transportation, Subchapters A, B, C

Bay West, Inc. Standard Operating Procedure, Field Decontamination at Nonradioactive Sites, CORP-ENV-002-65422V1, January 27, 2003.

Attachments

Uniform Hazardous Waste Manifest
Non-Hazardous Waste Manifest
Straight Bill of Lading

Please print or type. (Form designed for use on elite (12-pitch) typewriter.)

Form Approved. OMB No. 2050-0039

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator ID Number | 2. Page 1 of | 3. Emergency Response Phone | 4. Manifest Tracking Number 009688568 JJK

GENERATOR

5. Generator's Name and Mailing Address

Generator's Site Address (if different than mailing address)

Generator's Phone:

6. Transporter 1 Company Name | U.S. EPA ID Number

7. Transporter 2 Company Name | U.S. EPA ID Number

8. Designated Facility Name and Site Address | U.S. EPA ID Number

Facility's Phone:

9a. HM	9b. U.S. DOT Description (including Proper Shipping Name, Hazard Class, ID Number, and Packing Group (if any))	10. Containers No.	10. Containers Type	11. Total Quantity	12. Unit Wt./Vol.	13. Waste Codes
	1.					
	2.					
	3.					
	4.					

14. Special Handling Instructions and Additional Information

15. **GENERATOR'S/OFFEROR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labeled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations. If export shipment and I am the Primary Exporter, I certify that the contents of this consignment conform to the terms of the attached EPA Acknowledgment of Consent.
I certify that the waste minimization statement identified in 40 CFR 262.27(a) (if I am a large quantity generator) or (b) (if I am a small quantity generator) is true.

Generator's/Offeror's Printed/Typed Name | Signature | Month | Day | Year

INT'L

16. International Shipments ☐ Import to U.S. ☐ Export from U.S. Port of entry/exit:

Transporter signature (for exports only): Date leaving U.S.:

TRANSPORTER

17. Transporter Acknowledgment of Receipt of Materials

Transporter 1 Printed/Typed Name | Signature | Month | Day | Year

Transporter 2 Printed/Typed Name | Signature | Month | Day | Year

DESIGNATED FACILITY

18. Discrepancy

18a. Discrepancy Indication Space ☐ Quantity ☐ Type ☐ Residue ☐ Partial Rejection ☐ Full Rejection

Manifest Reference Number:

18b. Alternate Facility (or Generator) | U.S. EPA ID Number

Facility's Phone:

18c. Signature of Alternate Facility (or Generator) | Month | Day | Year

19. Hazardous Waste Report Management Method Codes (i.e., codes for hazardous waste treatment, disposal, and recycling systems)

1.	2.	3.	4.

20. Designated Facility Owner or Operator: Certification of receipt of hazardous materials covered by the manifest except as noted in Item 18a

Printed/Typed Name | Signature | Month | Day | Year

NON-HAZARDOUS WASTE MANIFEST

1. Generator ID Number	2. Page 1 of	3. Emergency Response Phone	4. Waste Tracking Number

GENERATOR

5. Generator's Name and Mailing Address

Generator's Site Address (if different than mailing address)

Generator's Phone:

6. Transporter 1 Company Name — U.S. EPA ID Number

7. Transporter 2 Company Name — U.S. EPA ID Number

8. Designated Facility Name and Site Address — U.S. EPA ID Number

Facility's Phone:

	9. Waste Shipping Name and Description	10. Containers No.	10. Containers Type	11. Total Quantity	12. Unit Wt./Vol.	
	1.					
	2.					
	3.					
	4.					

13. Special Handling Instructions and Additional Information

14. **GENERATOR'S/OFFEROR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name, and are classified, packaged, marked and labeled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Generator's/Offeror's Printed/Typed Name — Signature — Month Day Year

INT'L

15. International Shipments ☐ Import to U.S. ☐ Export from U.S. Port of entry/exit:

Transporter Signature (for exports only): Date leaving U.S.:

TRANSPORTER

16. Transporter Acknowledgment of Receipt of Materials

Transporter 1 Printed/Typed Name — Signature — Month Day Year

Transporter 2 Printed/Typed Name — Signature — Month Day Year

DESIGNATED FACILITY

17. Discrepancy

17a. Discrepancy Indication Space ☐ Quantity ☐ Type ☐ Residue ☐ Partial Rejection ☐ Full Rejection

Manifest Reference Number:

17b. Alternate Facility (or Generator) — U.S. EPA ID Number

Facility's Phone:

17c. Signature of Alternate Facility (or Generator) — Month Day Year

18. Designated Facility Owner or Operator: Certification of receipt of materials covered by the manifest except as noted in Item 17a

Printed/Typed Name — Signature — Month Day Year

STRAIGHT BILL OF LADING

ORIGINAL — NOT NEGOTIABLE

Shipper No. _____

Carrier No. _____

Page _____ of _____

_____ (Name of carrier) (SCAC)

Date _____

On Collect on Delivery shipments, the letters "COD" must appear before consignee's name or as otherwise provided in Item 430, Sec.1.

TO:

Consignee _____

Street _____

City _____ State, _____ Zip Code _____

FROM:

Shipper _____

Street _____

City _____ State _____ Zip Code _____

24 hr. Emergency Contact Tel. No. _____

Route _____

Vehicle Number _____

No. of Units & Container Type	HM	BASIC DESCRIPTION Proper Shipping Name, Hazard Class UN or NA Number, Packing Group or UN or NA Number, Proper Shipping Name, Hazard Class, Packing Group	TOTAL QUANTITY (Weight, Volume, Gallons, etc.)	WEIGHT (Subject to Correction)	RATE	CHARGES (For Carrier Use Only)

PLACARDS TENDERED: YES ☐ NO ☐

Note — (1) Where the rate is dependent on value, shippers are required to state specifically in writing the agreed or declared value of the property, as follows: "The agreed or declared value of the property is hereby specifically stated by the shipper to be not exceeding _____ per _____."
(2) Where the applicable tariff provisions specify a limitation of the carrier's liability absent a release or a value declaration by the shipper and the shipper does not release the carrier's liability or declare a value, the carrier's liability shall be limited to the extent provided by such provisions. See NMFC Item 172.
(3) Commodities requiring special or additional care or attention in handling or stowing must be so marked and packaged as to ensure safe transportation. See Section 2(e) of Item 360, Bills of Lading, Freight Bills and Statements of Charges and Section 1(a) of the Contract Terms and Conditions for a list of such articles.

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked and labelled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

_____ Signature

REMIT C.O.D. TO:
ADDRESS

COD Amt: $

C.O.D. FEE:
PREPAID ☐
COLLECT ☐ $

Subject to Section 7 of the conditions, if this shipment is to be delivered to the consignee without recourse on the consignor, the consignor shall sign the following statement:
The carrier shall not make delivery of this shipment without payment of freight and all other lawful charges.

_____ (Signature of Consignor)

TOTAL CHARGES $

FREIGHT CHARGES
FREIGHT PREPAID except when box at right is checked ☐ Check box if charges are to be collect

RECEIVED, subject to the classifications and tariffs in effect on the date of the issue of this Bill of Lading, the property described above in apparent good order, except as noted (contents and condition of contents of packages unknown), marked, consigned, and destined as indicated above which said carrier (the word carrier being understood throughout this contract as meaning any person or corporation in possession of the property under the contract) agrees to carry to its usual place of delivery at said destination, if on its route, otherwise to deliver to another carrier on the route to said destination. It is mutually agreed as to each carrier of all or any of, said property over all or any portion of said route to destination and as to each party at any time interested in all or any said property, that every service to be performed hereunder shall be subject to all the bill of lading terms and conditions in the governing classification on the date of shipment.

Shipper hereby certifies that he is familiar with all the lading terms and conditions in the governing classification and the said terms and conditions are hereby agreed to by the shipper and accepted for himself and his assigns.

SHIPPER _____ CARRIER _____

PER _____ PER _____

DATE _____

1

Permanent post-office address of shipper.

Bay West

Standard Operating Procedure

Ground Water Sampling

CORP-ENV-009-1510481

Revised: February 10, 2012

Review and Approval:

Developed by: _____
Signature
QA/QC Manager
Title
Date: 02-17-2012

Developed by: _____
Signature
QA/QC Manager
Title
Date: 02-17-2012

Approved by: _____
Signature
Vice President of Operations
Title
Date: 02/20/2012

Questions and requests for information regarding this SOP should be directed to the Vice President of Operations or the QA/QC Manager. This SOP cannot be edited, changed, or revised without the approval of the individuals listed above, and all edits, changes, and revisions must be routed through the Document Management Coordinator.

TABLE OF CONTENTS

1.0 INTRODUCTION

1.1 Purpose

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures employed by Bay West, Inc. to collect representative water samples from monitoring wells and water supply wells.

2.0 RESPONSIBILITIES

Sampler - The Sampler is responsible for the sampling, collection, labeling, analysis and recording of data as specified in this SOP. The Sampler is also responsible for the daily calibration of the field equipment.

Shop Manager - The Shop Manager is responsible for the maintenance of all monitoring equipment used by the company. The sampler is responsible for reporting all equipment problems to the Shop Manager for resolution.

Project Manager - The Project Manager is responsible for maintaining logbooks and forms and for approving techniques not specifically described in this SOP. The Project Manager must also document and initiate a corrective action investigation as appropriate when laboratory quality assurance sample results or other data indicate water samples may not be fully representative of the sampling location.

3.0 REQUIRED EQUIPMENT

This section provides a list of equipment to be used but does not necessarily include all equipment that will be used. Bay West SOPs shall be obtained and followed for decontamination, sampling, packaging and shipping. Specific SOPs are listed below:

- Field Equipment Decontamination at Non-Radioactive Sites (CORP-ENV-002-65422V1). Presents procedures for decontamination of field sampling equipment. All non-disposable equipment will be decontaminated between sampling locations to prevent cross contamination of monitoring wells.
- Packaging and Shipping of Environmental Samples (CORP-ENV-006-1510206). Establishes packaging and shipping requirements and guidelines for environmental sample shipping.
- Sample Custody (CORP-ENV-004-1510208). Presents sample custody procedures to maintain and document sample possession that are traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings.
- Field Documentation (CORP-ENV-010-41712V1). Presents the requirements for documenting field operations. The reader should be able to reconstruct in their

mind what took place in the field that day by reading the details of the field documentation from the site.

The following subsections identify the types of equipment along with equipment specifications. Prior to initiating field work it will be necessary to determine the type of equipment that will be needed to complete the sampling. In addition to the equipment specified in the following sections, the sampler will also obtain the following materials before initiating field work:

- Field log book
- Field Sampling Data Sheets
- Equipment manuals

3.1 Water/Level Sensors

Static water level measurements will be collected with an electronic water level indicator capable of reading in increments of 0.01 feet. In cases where free-phase hydrocarbon liquid is anticipated to be present in the well, an oil/water interface probe will be used to collect "static" product and water level measurements.

3.2 Monitoring Well Purging Equipment

There are several types of monitoring well purge equipment that can be used at a site. Equipment types and recommended use are provided in the Table 3.2-1 and briefly discussed below. Variables, such as purge volume, weather, accessibility, etc., may necessitate the use of alternative equipment.

Collecting volatile organic samples require special considerations when choosing a pump. Submersible pumps, such as the Grundfos Reddieflow or the Whalen pump contain impellers which drive the water to the surface. These impellers may cause cavitation during operation and de-gas some of the volatiles from the water phase. In addition, these pumps tend to heat up during prolonged periods of operation.

Peristaltic pumps, which produce a vacuum in the line to draw water to the surface, can cause de-gassing of some of the volatiles in the water. A bladder pump is the pump of choice when sampling for volatile organic compounds.

Table 3.2-1

Purge Equipment	Situation
Submersible Pump	High yield monitoring wells with a depth to water > 25-feet.
Bladder Pump	High yield monitoring wells with a depth to water > 25-feet.
12 Volt Whalen Pump	Single stage - High yield monitoring wells with a depth to water < 25-feet.
	Two stage - High yield monitoring wells with a depth to water > 25-feet but less than 60-feet.
Peristaltic Pump	Low yield monitoring wells with a depth to water < 25-feet.
Bailer	Low and high yield monitoring wells with limited access.

The submersible pump will be a Grundfos Reddieflow or equivalent with 110 V AC MP-1 controller. The low flow pump will be a QED low flow bladder pump with a controller powered by breathing air or an oilless air compressor. The Whalen pump can be used with a 12 volt battery. The peristaltic pump can be used with a 12 volt battery or a built-in 110 volt power converter.

3.3 Stabilization Equipment

There are several types of stabilization equipment that can be used at a site. Equipment names, model numbers, and parameters measured are provided in Table 3.3-1. Bay West owns several YSI 650/6820 Multi-Meters flow through cells for groundwater sampling. These meters will measure all of the required parameters simultaneously. The parameter tolerances that will be used for Bay West projects, unless modified in site-specific sampling plans, are specified in Section 4.2 (EPA, 1996, Appendix B).

Table 3.3-1

Stabilization Equipment	Measures
YSI Dissolved Oxygen/Temperature Meter, Model 55	Dissolved Oxygen (DO) and Temperature
YSI Specific Conductance/Temperature Meter, Model 33 SCT	Specific Conductance and Temperature
Orion, pH/Temperature Meter, Model SA210	pH and Temperature
YSI Multi-Meter with flow-through cell, Model 650/6820	DO, Specific Conductivity, Oxidation/Reduction Potential (ORP), Temperature, pH, and Turbidity

Calibration and maintenance procedures for the YSI 650/6820 Multi-Meter are included in Appendix C.

3.4 Vacuum Filtration Equipment

Filtration equipment may be used to filter samples for metals analysis. The following equipment will be used for filtering water samples:

- Peristaltic pump, bladder pump, submersible pump
- QED® 0.45-micron, disposable, in-line filter

These QED® filters are for single use only and a new filter will be used for each monitoring point and disposed after each use. After attaching the filter to the pump, rinse the filter with 50-100 ml of water before collecting the sample.

3.5 Sample Containers

Sample containers will be provided by the laboratory conducting the analysis. Sample bottles will be new glass containers with Teflon®-lined lids. Sample containers will be chosen according to U.S. EPA analytical guidelines.

4.0 PROCEDURES

Ground water monitoring and sampling will be performed in general accordance with standard industry practices. This section is organized in the order that a specific task or objective will be performed in the field. All ground water monitoring data will be recorded on the Field Sampling Data Sheet (Appendix A) and/or site log books. Trip blanks, field blanks, and duplicate samples will also be collected as outlined in Section 6.2 of this SOP.

4.1 Water/Level Sensors

Prior to well purging or sampling, the initial depth to static water elevation will be measured at each well with a water level indicator or oil/water interface probe to the nearest 0.01-feet. The sampler will make water level measurements at all appropriate monitoring wells and piezometers within the shortest time interval practical to provide comparable numbers by which to calculate the ground water gradient. Static depth measurements will be measured relative to the top, inside edge (TOC) of the monitor well casing. Monitor wells that do not bear a TOC benchmark will be measured from the north side of the casing and a TOC benchmark will be added.

The total completion depth of the monitor well will be measured relative to the TOC and the bottom of the well screen. Monitor well casing volumes will subsequently be determined by the following calculation:

Casing Volume (gallons) =
[Total Depth (feet) - Static Depth (feet)] * [Well Diameter^2 (inches)] * (0.0408)

The measurements will be collected by inserting the probe into the well until the water contact buzzer activates, at which point the static elevation value will be recorded. The probe will be raised above the air/water interface and a second measurement taken to verify the recorded value. Do not lower the probe to the bottom of the well at this time. Doing so will raise the turbidity of the water column and increase purging time. AFTER

sample collection is complete, then the total depth of the well may be measured, if required.

4.2 Monitoring Well Purging and Stabilization

Monitor well purging will be done by one of two approved methods (pumping or bailing). The UFP-QAPP will contain details on the method required for the site.

Monitoring wells will be purged in conjunction with stabilization testing. Generally, bailing will be performed from the top of the water column in the well. With the low flow pump, purging will be performed from the middle of the well screen.

Bailing Method:
Three to five well volumes of water will be purged from the top of the water column in the well and stabilization readings will be taken at a minimum of one set of readings per well volume. Additional volumes may be required by state regulations or site conditions. Consult the site specific Sampling and Analysis Plan (SAP) for the job requirements.

Pumping Method (Low-Flow preferred)
Stabilization readings are taken every three to five minutes until stabilization is reached. The pump rate during purging must be <1 L/minute and during sampling must be <300 ml/minute. Care should be taken to avoid drawing down the water column in the well.

The stabilization readings will generally be taken using a closed flow-through cell. The stabilization parameters will be recorded on the Field Sampling Data Sheet and/or the field log book every 3-5 minutes during low flow purging. If the bailing method is being employed, the readings are recorded after the purging of each well volume until the values of the parameters are within the following tolerances on three successive readings (low flow or bailing):

- Temperature +/- 1 ºC
- pH +/- 0.1 standard units
- Specific conductance +/- 3% or +/- 0.01 mS/cm
- Turbidity – 10% ideally , sampling may commence when the readings stabilize ≤10 NTUs (Recommended if metals samples will not be filtered)
- DO +/- 10% or +/- 0.3 mg/L (whichever is less)
- ORP +/- 10 mv or 10% (whichever is less)

If a flow through cell is not used, water will be transferred from the monitoring well using a pump or bailer to the sample chamber of the meter where the pH, temperature, DO, ORP and specific conductance may be measured.

During the stabilization testing, the following data will be recorded on the Field Sampling Data Sheet:

- Time
- Parameters listed above and the values measured
- Cumulative volume purged.

In addition to the above parameters, color and odor will be described and recorded on the Field Sampling Data Sheet and/or log book.

The cell discharge line will deliver the water stream to a temporary storage container. The water from sources known to be non-contaminated will be returned to the environment. Purge water from contaminated sources will be collected, stored, tested, and disposed of in accordance with site-specific project plans and applicable state and federal regulations.

4.3 Vacuum Filtration Equipment

New filter units will be used at each monitoring well and disposed after use. The filter unit inlet will be fitted to the sample discharge line of the pump after well stabilization. The pump will supply the back pressure required for filtration. When bailers are used to collect the sample, a clean jar with no preservative is filled with the sample water to be filtered. The water is then passed through a peristaltic pump attached to the filter inlet. The sample container will be filled directly from the filter unit outlet.

4.4 Sample Collection and Containers

Ground water samples from monitoring wells, unless otherwise specified, will be collected after well stabilization. Samples will be collected using disposable bailers or through the discharge tubing from the purging pump
A clean and dry sheet of relatively inert plastic shall be placed on the ground surface in the wellhead area. If materials used in the sampling process must be put down they will be placed on a clean portion of the plastic sheet, not the ground surface.

The bailer will be manually suspended over the monitoring well (downrigger may be used; the downrigger will be pre-cleaned with a deionized water rinse). New disposable nitrile gloves will be worn when handling the bailer and discarded after each well. To collect the ground water sample, the bailer will be lowered into the monitoring well until it is submerged below the top of the water column. Once the bailer has been filled, the bailer will be retrieved to the top of the monitoring well. The bottom unloading device will be inserted into the ball check valve until the water is flowing from the bailer. The sample container will be filled from the unloading device.

For wells stabilized via the low flow method, all samples will be collected from the discharge tubing of the pump. After disconnecting the flow-through cell from the discharge tubing, the flow rate may be reduced slightly to aid in filling the bottles. Care

should be taken not to overfill or splash the preservatives out of the bottles while filling. Do not collect any water that has gone through the cell.

Sample Containers shall be filled in the following order:

- Major and Minor ions
- Nitrates
- Cyanide
- Trace metals
- Chromium VI
- Miscellaneous parameters
- Volatile Organics (VOCs)
- Non-volatile organics (SVOCs, Pesticides, PCBs, Herbicides)
- Dioxin and dibenzo furans
- Coliform bacteria
- Total Organic Carbon (TOC)
- Total phosphorus
- Sulfide
- Radium, gross alpha, and gross beta

When sampling for volatile organics, the sample vials will be slightly overfilled to achieve a positive meniscus at the mouth of the bottle. Sealing the VOC bottle with the septum top cap should prevent any air bubbles from forming. Samples will not be filtered unless site-specific project requirements specify filtration. Samples will not be transferred from one container to another.

Samples to be analyzed will be preserved using pre preserved glassware provided by the analytical lab doing the analysis. Additional preservation information can be found in the documents referenced in Section 7.0.

If necessary, the sampler will use their body to shield the sampling container from wind and airborne dust while filling sample containers. When strong winds, heavy rain, or dusty conditions are present, additional measures will be implemented to guard against background interference.

4.5 Water Supply Sample Collection from a Tap

Water supply samples will be collected manually from a tap upstream of any treatment equipment. Sample bottles will be filled directly from the tap, if possible. Otherwise, using a clean, spare, unpreserved glass sample jar, transfer the water to the sample container being sent to the lab. The water will be flushed through the tap for a minimum of 10 to 15 minutes prior to sample collection.

When the SAP requires sampling for biological parameters (e.g. Coliform bacteria) from a tap, the outside surface of the tap opening will be sterilized prior to sampling. A match or a lighter shall be used to quickly heat the tap opening immediately prior to sample collection. Be sure to extinguish all heat sources properly after use.

5.0 DOCUMENTATION

All information will be documented on the Field Sampling Data Sheet and/or the Field Log Book. If any editing of field logbook is required, the erroneous entry will be crossed out with a single line and dated and initialed. If a log book is used to record field stabilization parameters, the readings must be transferred within 24 hours to a Field Sampling Data Sheet. A Field Sampling Data Sheet will be completed for each source sampled. The source of any reference material or field charts used must also be recorded in the field logbook. Refer to the Field Documentation SOP (CORP-ENV-010-41712V1).

The following is a general summary of the information to be documented:

- List of all personnel present
- Field conditions including:
    - Air temperature
    - Wind speed/direction
    - Precipitation/moisture at the time of sampling event
    - Ambient odors
    - Airborne dust
- Description of any exceptions to this SOP or the SAP
- For each well sampled:
    - Well Name and unique number used to identify the sample
    - Equipment used for purging and stabilization
    - Date and time that purging and sampling began and ended
    - A list of all samples sent to each laboratory
    - Complete a Field Sampling Data Sheet to record all purging and stabilization data

6.0 PERFORMANCE EXPECTATIONS

6.1 Restrictions/Limitations

Only qualified persons trained in performing the requirements and the duties described in the SOP shall conduct the work. All Bay West sampling technicians will have a

minimum of two years education or experience in environmental sampling. In addition, sampling technicians will comply with the safety training and medical surveillance requirements of 29 CFR 1910.120. The Project Manager will have the authority to decide whether or not an individual is qualified.

6.2 Quality Assurance/Quality Control

Quality Assurance/Quality Control (QA/QC) samples will consist of field blanks (FB), trip blanks (TB), temperature blanks and duplicate samples.

Field Blank - A field blank is a sample of Deionized Water (DI) poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. Field blanks are used to assess the effectiveness of Bay West equipment decontamination procedures. If laboratory results for a field blank exceed one-half of the RLs for the project, the Project Manager must document and initiate a corrective action investigation.

The frequency of collection for field blanks is typically one per day of sampling however, site-specific project plans may specify otherwise. Field blanks shall be collected immediately after the equipment has been decontaminated. The field blank is typically analyzed for all laboratory analyses requested for the environmental samples collected at the site.

Trip Blank - The Trip Blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample, and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler of samples sent to the laboratory for analysis of VOCs shall contain one trip blank unless otherwise specified in the site-specific project requirements.

When an analyte is detected in the trip blank at concentrations exceeding one half of the RLs for the project, the Project Manager must document and initiate a corrective action investigation.

Temperature Blank - The temperature blank consists of a plastic bottle filled in the laboratory with water, and placed in sample coolers before or during sample collection to ensure temperature equilibration with the samples and returned to the laboratory. Upon receipt of samples, the laboratory will determine and record the temperature of the samples using the temperature blank.

Field Duplicates - A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The frequency of collection for duplicate samples is determined by the project manager based on site-specific project requirements. The sample containers are assigned a unique identification number in the field to ensure the duplicate is processed as a "blind" duplicate from the laboratory's perspective. Specific locations may be designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection and analytical processes.

Bay West SOPs shall be followed for decontamination of all sampling equipment (CORP-ENV-002-65422V1), and sampling, packaging and shipping of all environmental samples (CORP-ENV-006-1510206).

7.0 REFERENCES

U.S. EPA, Groundwater Issue, Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures, EPA/540/S-95/504, April 1996. (EPA, 1996)

USACE, Geology Supplement to the Scope of Services, Revised May 2011

Manuals for each equipment type specified in Section 3.0.

Minnesota Pollution Control Agency, Groundwater Unit, Field Guidance Manual, July, 1998.

8.0 APPENDICES

APPENDIX A Field Sampling Data Sheets DOCs #119471

APPENDIX B U.S. EPA, Groundwater Issue, Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures

APPENDIX C Calibration and Maintenance Procedures for the YSI 650/6820 Multi-Meter for Low Flow Sampling

Appendix D

Field Forms

- Daily Construction Quality Control Report
- HTRW Drilling Log
- Soil Sample Collection Field Sheet
- Water Sample Collection Field Sheet

Bay West

DAILY CONSTRUCTION QUALITY CONTROL REPORT

Contract No.: W9128F-10-D-0025, DO # 0002 **Date: _____Rpt. No.: ___**
Project Title & Location:_____

Weather: Clear____ Partly Cloudy ___ Cloudy ___ Rainfall __ (__% of workday)
Temperature during workday: High ___ degrees F. Low ____ degrees F.

1. WORK PERFORMED BY CONTRACTORS/SUBCONTRACTORS

Contractor	No. of Workers	Crafts	Hours	Description of Work

2. OPERATING EQUIPMENT DATA (Not hand tools)

Equipment	Date of arrival /departure	Owned or Rented	Hours Used	Hours Idle	Hours of Rep./Main

3. WORK PERFORMED TODAY: (Indicate location and description of work performed by prime and/or subcontractors).

4. QUALITY CONTROL INSPECTIONS & RESULTS (Include a description of preparatory, initial, and/or follow-up inspections or meetings; check of subcontractors work and materials delivered to site compared to submittals and/or specifications; comments on proper storage of materials; include comments on corrective actions to be taken):

5. QUALITY CONTROL TESTING AND RESULTS (comment on tests and attach test reports)
6. DAILY SAFETY INSPECTIONS (Include comments on new hazards to be added to Hazard Analysis and corrective action of any safety issues):
7. REMARKS (Include conversations with or instructions from the Government representatives; delays of any kind that are impacting the job; conflicts in the contract documents; comments on change orders; environmental considerations; etc.):

8. CONTRACTOR'S VERIFICATION: I certify that to the best of my knowledge the above report is complete and correct. All material, equipment used, and work performed during this reporting period is in compliance with the contract plans and specifications except as noted above.

_____

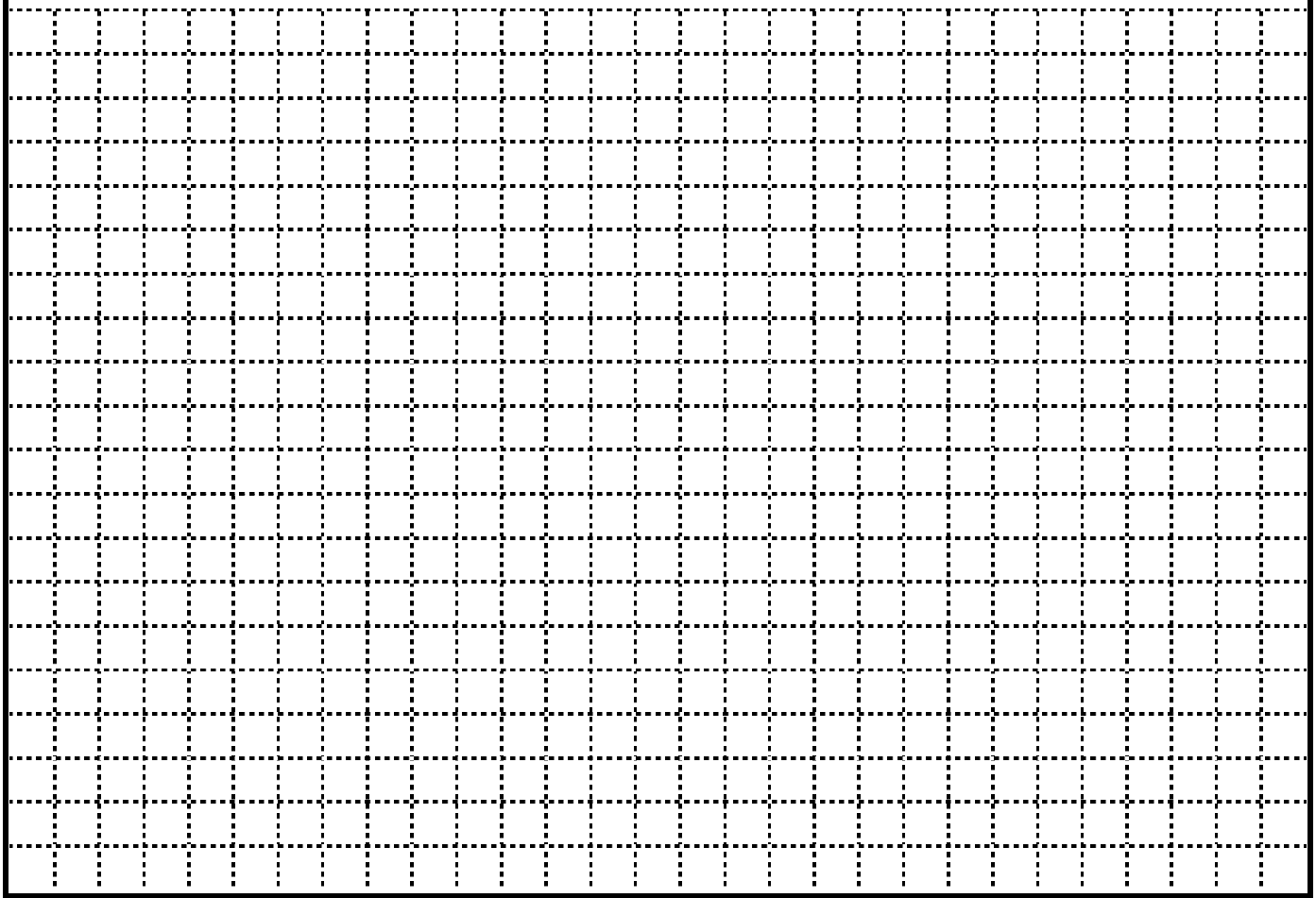
Contractor Quality Control Officer

HTRW DRILLING LOG

	DISTRICT	HOLE NUMBER
1. COMPANY NAME	2. DRILLING SUBCONTRACTOR	SHEET SHEETS OF
3. PROJECT	4. LOCATION	
5. NAME OF DRILLER	6. MANUFACTURER'S DESIGNATION OF DRILL	
7. SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT	8. HOLE LOCATION	
	9. SURFACE ELEVATION	
	10. DATE STARTED	11. DATE COMPLETED
12. OVERBURDEN	15. DEPTH GROUNDWATER ENCOUNTERED	
13. DEPTH DRILLED INTO ROCK	16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING	
14. TOTAL DEPTH OF HOLE	17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES			
20. SAMPLES FOR CHEMICAL	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21. TOTAL CORE RECOVERY %
22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR		

LOCATION SKETCH/COMMENTS SCALE:



PROJECT	HOLE

HTRW DRILLING LOG (CONTINUATION SHEET)

HOLE

PROJECT

Inspector

SHEET SHEETS OF

ELEV. a.	DEPTH b.	DESCRIPTION OF MATERIALS c.	FIELD SCREENING RESULTS d.	GEOTECH SAMPLE OR COREBOX NO. e.	ANALYTICAL SAMPLE NO. f.	BLOW COUNTS g.	REMARKS h.

Project

HOLE NO.

ENG FORM 5056-R, AUG 94

(Proponent: CECW-EG)

SOIL SAMPLE COLLECTION FIELD SHEET

GENERAL INFORMATION

SITE NAME: _____ PROJECT NO. _____

SAMPLE NO. _____ BORING NO. _____

DATE/TIME COLLECTED: _____ PERSONNEL: _____

SAMPLE METHOD / DEPTH: _____

SAMPLE MEDIA: SOIL SEDIMENT SLUDGE

SAMPLE QA SPLIT: YES NO SPLIT SAMPLE NO. _____

SAMPLE QC DUPLICATE: YES NO DUPLICATE SAMPLE NO. _____

MS/MSD REQUESTED: YES NO

SAMPLE CONTAINERS, PRESERVATIVES, ANALYSIS

Sample Container	Preservative	Analysis Requested

OVA MEASUREMENTS

Background _____

Breathing zone _____

Boring _____

Headspace _____

SAMPLE DESCRIPTION

DEPTH:	DESCRIPTION:

GENERAL COMMENTS

WATER SAMPLE COLLECTION FIELD SHEET

GENERAL INFORMATION

SITE NAME _____ PROJECT NO. _____

SAMPLE NO. _____ WELL NO. _____

DATE/TIME COLLECTED _____ PERSONNEL _____

SAMPLE METHOD _____

SAMPLE MEDIA:	Groundwater	Surface Water	
SAMPLE QA SPLIT:	YES	NO	SPLIT SAMPLE NO. _____
SAMPLE QC DUPLICATE:	YES	NO	DUPLICATE SAMPLE NO. _____
MS/MSD REQUESTED	YES	NO	

SAMPLE CONTAINERS, PRESERVATIVES, ANALYSIS

Sample Container	Preservative	Analysis Requested

WELL PURGING DATA

Date _____ Well Depth (ft. BTOC) _____

Time Started _____ Depth to Water (ft BTOC) _____

Time Completed _____ Water Column Length _____

Hnu Measurements Volume of Water in Well (gal) _____

Background _____ Casing Volumes to Purge _____

Breathing Zone _____ Minimum to Purge (gal) _____

Well Head _____ Actual Purge (gal) _____

FIELD MEASUREMENTS

Time	Amount Purged (gal)	pH	Temperature (ºC)	Conductivity (mS/cm)	Dissolved Oxygen (mg/L)	ORP (mV)	Turbidity (NTU's)

FIELD EQUIPMENT AND CALIBRATION

	Model	Calibration
Water Level Probe		
Water Quality Meter		

GENERAL COMMENTS

Appendix E

Laboratory Forms and Standard Operating Procedures

- Diesel and Residual Range Organics (DRO and RRO) by GC/FID (SW846 Method 8015C) Revision 2, 03/25/2011
- Gasoline Range Organics (GRO) by GC/FID (SW846 Method 8015C) Revision 7.1, 07/29/2011
- Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 Method 8082A) Revision 0.1, 06/11/2010
- Microwave Extraction of Solid Samples (SW-846 3546) Revision 1, 01/13/2011
- Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS, Method 8290, 8290A & TO-9A, Revision 7.3, 12/30/2009
- Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS Methods 8290, 8290A & TO-9A Revision 1.1, 02/05/2010
- TOC in Soil, Revision 0, 05/10/11

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

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Title: Diesel and Residual Range Organics (DRO and RRO) by GC/FID

[SW846 Method 8015 and others]

Approvals (Signature/Date):

Dennis Jonsrud — Date
GC Supervisor

3-30-12

Adam Alban — Date
Health & Safety Manager / Coordinator

Adam W Alban 02 April 12

John Morris — Date
Quality Assurance Manager

3/30/12

Robert C. Hanisch — Date
Laboratory Director

Robert C Hanisch 4/2/12

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure is designed to measure the concentration of diesel range organics (DRO), jet fuels, and motor oils in water or solid samples by EPA Methods 8015B, 8015C, 8015D, using method 8000B and 8000C criteria. Also addressed in this SOP are Alaska Methods AK102 and AK103, NWTPH (Washington) and the California LUFT method. The Oklahoma method appears in the appendix.

1.2 As most commonly defined, this corresponds to a carbon range of C_{10} – C_{36} (boiling point range of approximately 170 °C to 430 °C). This carbon range can include kerosene, several types of jet fuel, several types of diesel fuels, and a number of types of light heating oils. Other petroleum hydrocarbon ranges, any within the C_8 – C_{38} general range, may be characterized using this general method (details concerning the calibration and aliphatic reference standards need to be specified in the project work plan, and the final report must clearly indicate the range used).

1.3 Alaska Method AK102 for the determination of diesel range organics defines DRO as a carbon range from the beginning of C_{10} to the beginning of C_{25} (boiling point range of approximately 170 °C to 400 °C). Alaska method AK103 for the determination of residual range organics (RRO) has also been incorporated into this SOP. Alaska Method AK103 defines RRO as a carbon range from the beginning of C_{25} to the end of C_{36} (boiling point range of approximately 400 °C to 500 °C). It should be noted that this is essentially the same range as defined for motor oil.

1.4 Refer to Table 1 for boiling point information for the aliphatic hydrocarbons.

1.5 Petroleum products such as lubricating oils, waxes, and asphalt can have a significant fraction of higher molecular weight / higher boiling point components that are not detectable under the conditions of this method.

1.6 Analytes, Matrix(s), and Reporting Limits

1.6.1 The standard diesel reporting limit for this procedure is 0.1 mg/L for water samples and 4.0 mg/kg for soil samples. The jet fuel and motor oil reporting limits are 1 mg/L (0.5 mg/L for NWTPH) for water samples and 10 mg/kg for soil samples. Other reporting limits may apply to each of the listed methods with the level 1 initial calibration standard being the basis of the calibration for the lowest reporting limit.

1.6.2 Unless otherwise noted the most stringent criteria or common criteria will be reported and adhered to for the methods listed above.

1.6.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in SOP DV-QA-0031.

2.0 Summary of Method

2.1 The method involves extracting hydrocarbons from aqueous or solid samples with methylene chloride. See SOPs DV-OP-0006, DV-OP-0016, DV-OP-0015, and DV-OP-0007 for water sample extraction, solid sample extraction, and extract concentration procedures, respectively. For solvent-miscible products, medium/high level options are provided.

2.2 The methylene chloride extract is concentrated and analyzed with a gas chromatograph equipped with a flame ionization detector (GC/FID). Quantitation is performed by comparing the total peak area within the specified carbon range, both resolved and unresolved peaks, to the response of an authentic fuel or oil calibration standard.

3.0 Definitions

3.1 Diesel Range Organics (DRO): The sum of compounds producing chromatographic peaks, both resolved and unresolved, eluting from:

- the start of the n-decane peak (C_{10}) to the end of n-octacosane (C_{28}), or
- n-decane (C_{10}) to (beginning of for AK101) n-pentacosane (C_{25}), or
- n-decane (C_{10}) to n-docosane (C_{22}), or
- n-decane (C_{10}) to n-tetracosane (C_{24}), or
- n-decane (C_{10}) and n-hexatriacontane (C_{36}).

The markers used for integration for quantitation of DRO varies by method. The 8015 methods imply that the integration begins at the apex minus the RT window for the starting alkane and ends at the apex plus the RT window for the ending alkane marker. The Oklahoma method states that the integration begins 0.1 minutes before the initial alkane and ends 0.1 minutes beyond the ending alkane marker. NWTPH implies that the whole of the hydrocarbon pattern be incorporated into the range of integration (peaks at least down to 10% of the highest representative peak) and that the integration breaks where multiple hydrocarbon patterns are indicated for a single sample.

3.2 Diesel Calibration Standard: A retail-purchased diesel #2 or fuel oil #2 (the two products are essentially the same crude oil distillation cut) used as the calibration stock. The Oklahoma standard uses a ten component (C10-C28, even homologs) blend of alkanes.

3.3 Motor Oil (MO): The sum of the compounds producing chromatographic peaks, both resolved and unresolved, eluting from

- the start of the tetracosane (C_{24}) to n-hexatriacontane (C_{36}), or
- n-docosane (C_{22}) to n-hexatriacontane (C_{36}), or
- (the beginning of for AK103) n-pentacosane (C_{25}) to (the end of for AK103) n-hexatriacontane (C_{36}).

Alaska Method AK103 defines this latter range as “residual range organics.”

3.4 Jet Propellant-4 (JP-4): The range is determined by injecting a standard purchased from a vendor and choosing the retention times from the initial low point of the chromatographic peaks to the end of the resolved and unresolved peaks. The hydrocarbon range for this fuel is typically from C_4 to C_{12}.

3.5 Jet Propellant-8 (JP-8): The range is determined by the same method as used for JP-4. The hydrocarbon range for this fuel is typically from C_6 to C_{12}.

3.6 Residual Range Organics (RRO): All compounds producing chromatographic peaks, both resolved and unresolved, eluting between the peak start of n-pentacosance (C_{25}) and the peak end of n-hexatriacontane (C_{36}). See "motor oil" above (Section 3.3).

3.7 Boeing and Oklahoma: These carbon ranges and standards are defined and explained in detail in Appendix 1.

4.0 Interferences

4.1 Although primarily designed as a petroleum hydrocarbon test procedure, other organic compounds including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters, and biogenic terpenes are measurable under the conditions of this method. A silica gel cleanup can be used to remove these biogenic and substituted hydrocarbons. The details of the cleanup procedure are not given in this SOP and would have to be detailed in an approved project QAPP.

4.2 Heavier petroleum products such as lubricating oil and crude oil also produce a response within the retention time range for DRO. As defined in the method, the DRO results include these compounds.

4.3 Method interferences are reduced by washing all glassware in accordance with SOP DV-OP-0004. At least one method blank must be analyzed with each extraction batch to demonstrate that the samples are free from method interferences.

4.4 High-purity reagents must be used to minimize interference problems.

4.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for cross-contamination.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of these zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant Poison	25 ppm – TWA 125 ppm – STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm – TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit..			

6.0 Equipment and Supplies

6.1 Instrumentation

Gas Chromatograph: Analytical system including appropriate gas supply and all required accessories, including a Flame Ionization Detector (FID), column supplies, gases, and syringes. A data system capable of determining peak areas using a forced baseline and baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.

6.2 Supplies

6.2.1 Restek Corporation Rtx®-1 (fused silica), 30 m, 0.32 mm ID, 0.25 μm film thickness.

6.2.1.1 Other columns that meet the performance criteria stipulated in Section 10.0 may be used. Capillary columns are required to achieve the necessary resolution.

6.2.2 2-mL glass vials with Teflon-lined cap (autosampler vials).

6.2.3 40-mL VOA vials with Teflon-lined screw caps.

6.2.4 Y-splitter, septa, guard columns, ferrules, Siltek injection port liners, Siltek glass wool. Use Agilent liner 5183-4647 for instrument U2.

6.2.5 Microsyringes (µL): 1, 5, 10, 25, 100, 250, 500, 1000, and 5000 µL.

6.2.6 Volumetric flasks: 10.0, 50.0, and 100 mL, Class A glass.

6.2.7 An analytical balance capable of accurately weighing 0.0001 g must be used for preparing standards. A top-loading balance capable of weighing to the nearest 0.01 g must be used for sample preparation.

6.3 Computer Software and Hardware

6.3.1 Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Methylene chloride, pesticide grade or equivalent. Each lot must be approved for use in accordance with CA-Q-S--001.

7.1.2 Acetone, pesticide grade or equivalent. Each lot must be approved for use in accordance with CA-Q-S-001.

7.1.3 Ottawa sand (reagent sand) for solid matrix method blanks and lab control samples.

7.2 Standards

7.2.1 RT Reference Standard: DRO Mixture (Tennessee/Mississippi) and Calibration/Window Defining Hydrocarbon Standard. Current source Accu Std DRH-004S-R1-5x and Ultra Sci UST-210 both at 1000 ug/ml.

7.2.2 Stock Diesel Fuel Standard – Vendor purchased Diesel fuel oil #2 at a concentration of 50,000 µg/mL in methylene chloride. Current source Restek 31259.

7.2.3 Stock Motor Oil / Residual Range Organics (RRO) Standard – Equal weights of 30-weight and 40-weight motor oils are composited and used to make this standard. Alternatively, the Alaska method standard may be purchased from a commercial source as a stock solution in methylene chloride at a concentration of 50,000 µg/mL. Current source is Ultra Sci RGO-724.

7.2.4 Stock Jet Fuel Standards, JP8 and JP4 - Purchased from a vendor at concentrations of 10,000 µg/mL in methylene chloride.

7.2.5 Stock Surrogate Solution – Purchased as a custom mix from a vendor, consisting of ortho-terphenyl and n-octacosane, at a concentration of 5,000 μg/mL each in methylene chloride. Accu Std S-8087-2.5x.

NOTE: Alaska Method AK103 (RRO) requires the use of n-triacontane-d62 as a surrogate. However, the Alaska Certification Office has granted approval to use n-octacosane (C28) as the surrogate.

7.2.6 Surrogate Spike Solution – Dilute 1.0 mL of Stock Surrogate Solution to a final volume of 250 mL using 90:10 acetone : methylene chloride for a final concentration of each surrogate compound at 20 μg/mL.

7.2.7 Spike Solution – Spike solutions may be prepared as single or multi-component solutions in acetone as follows:

Standard Solution	Vol of Standard Solution (mL)	Final Vol (mL)	Conc (μg/mL)
Stock Diesel Fuel Standard	8.0	200.0	2,000
Stock Jet Fuel Standard (JP8)	1.0	10.0	1,000
Stock Jet Fuel Standard (JP4)	1.0	10.0	1,000
Stock RRO (or Motor Oil) Standard	1.0	10.0	5,000

7.2.8 Diesel Fuel Calibration Standards – Diesel fuel calibration standards are prepared in methylene chloride at 7 concentration levels using either the Stock Diesel Fuel Standard (Section 7.2.2) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Diesel Conc (μg/mL)	Surrogate Conc (μg/mL)
7	Stock	0.6	1.0	30,000	NA
6	Stock + Stock Surrogate	1.5 and 0.075	5.0	15,000	75.0
5	Level 6	0.5	1.0	7500	37.5
4*	Stock + Stock Surrogate	1 and 0.05	10	5000	25.0
3	Level 4	0.2	1.0	1000	5.0
2	Level 4	0.05	1.0	250	1.25
1	Level 4	0.02	1.0	100	0.5

NOTE: The Level 4 (*) calibration standard is also used as the continuing calibration verification (CCV) standard.

Only the diesel fuel calibration standards contain the surrogates.

7.2.9 Jet Fuel Calibration Standards – Jet Fuel (JF) Calibration standards are prepared in methylene chloride at 6 concentration levels using either the Stock Jet Fuel Standards (Section 7.2.4) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc (µg/mL)
6 JF	Stock	NA	NA	10,000
5 JF	Stock	0.125	0.250	5000
4 JF	Stock	0.0625	0.250	2500
3 JF*	Stock	1.0	10.0	1000
2 JF	Level 3 JF	0.125	0.250	500
1 JF	Level 3 JF	0.025	0.250	100

NOTE: The Level 3 JF (*) jet fuel calibration standard is used as the CCV standard.

7.2.10 Motor Oil / RRO Calibration Standards: Motor Oil (MO) (or Alaska Method RRO) calibration standards are prepared in methylene chloride at 7 concentration levels using either the Stock Motor Oil / RRO Standard (Section 7.2.3) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc (µg/mL)
7RRO	Stock	0.6	1.0	30,000
6RRO	7RRO	0.5	1.0	15,000
5 RRO	7RRO	0.250	1.0	7500
4 RRO*	Stock	0.5	5.0	5000
3 RRO	Level 4 RRO	0.2	1.0	1000
2 RRO	Level 4 RRO	0.05	1.0	250
1 RRO	Level 4 RRO	0.02	1.0	100

NOTE: The Level 4 MO (*) calibration standard is used as the CCV standard.

7.2.11 Diesel Fuel Continuing Calibration Verification Standard (CCV): The Level 4 Diesel Fuel Calibration Standard (Section 7.2.8) is used as the CCV standard.

7.2.12 Jet Fuel CCV Standard: The Level 3 JF jet fuel calibration standard (Section 7.2.9) is used as the jet fuel CCV standard.

7.2.13 Motor Oil / RRO CCV Standard: The Level 4 MO motor oil / RRO calibration standard (Section 7.2.10) is used as the motor oil / RRO CCV standard.

7.2.14 Second-Source Calibration Verification Standards: A standard containing diesel fuel or motor oil/RRO in methylene chloride at a concentration of 1,000-µg/mL and jet fuel at a concentration of 2,500 µg/mL is used to verify each initial

calibration. This standard uses diesel fuel, jet fuel and motor oil obtained from a source different than the source for the primary standard.

7.2.15 The standards listed above may also be prepared from certified commercial sources, in which case, the starting stock standards may be at lower concentrations. One-time deviations from the directions shown above must be recorded in the standards dilution log.

7.3 Standards Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.4 Storage of Stock Standards

Stock standard solutions are stored per vendor recommendations, generally at room temperature. Working level standard solutions are kept refrigerated. Standard solutions must be replaced every six months or sooner as specified by the vendor. Standard solutions must be replaced more frequently if comparison with check standards indicates a problem.

7.5 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.5.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.3 above.

7.5.2 Non-routine standards will be stored and treated as described in Section 7.4 above or as specified by the manufacturer.

7.5.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.5.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.5.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in duplicate, in 1-liter amber glass containers with Teflon-lined screw caps. Alaska Method AK102 requires that the water sample be acidified to a pH of 2 using hydrochloric acid.

8.2 Soils are collected in a core tube, or 4- or 8-ounce glass jar with Teflon-lined lid. The samples are stored at ≤ 6 °C from the time of collection until extraction.

8.3 Extraction must be performed on water samples within 7 days, and soil samples within 14 days. Methods AK102, AK103, and NWTPH allow a 14 day holding time for water extraction when preserved. All analyses must take place within 40 days from extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Demonstration of Capability

9.2.1 Before analyzing samples, the analyst must demonstrate the ability to operate the instrumentation and generate data that meets method criteria (IDOC). The analyst must also establish a method detection limit (MDL) for each instrument used for a particular method of analysis. The initial demonstration and method detection limit (MDL) studies (see Policy DV-QA-005P) must be acceptable before analysis of samples may begin. An MDL verification standard prepared at approximately two times the MDL is analyzed quarterly to demonstrate the qualitative reliability of the calculated MDL value. MDLs are verified annually or when major changes are made to the analytical processes. Likewise ongoing proficiency must be demonstrated by each analyst on an annual basis. MDLs are

stored in the LIMS. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Sample QC

The following quality control samples are prepared with each batch of samples.

9.3.1 Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed together using the same reagents and standards. Each quality control batch must contain a method blank (MB), a laboratory control sample (LCS), and matrix spike - matrix spike duplicate (MS/MSD) pair. If there is not enough sample volume for an MS/MSD then a different duplicate LCS may be used to determine precision. For more details see Policy DV-QA-003P.

NOTE: The AK-102 and AK-103 Methods require duplicate LCSs (i.e., LCS and LCSD) for each batch. The NWTPH and Oklahoma methods specify that duplicates be supplied every 10 samples.

9.3.2 Method Blank (MB)

One method blank is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The method blank consists of either 1 liter of organic-free water (for batches of aqueous samples) or 30 grams of Ottawa sand (for batches of soil samples). Method AK102 and 103 specifies a 25 gram sample size for soils. Oklahoma and NWTPH methods specify a 20 gram soil sample size. The method blank is processed exactly as samples in the batch, and is used to assess whether the laboratory processes have contaminated the samples in the batch.

Acceptance Criteria: Results for the method blank must be less than or equal to one-half the reporting limit concentration or less than 10% of the lowest concentration found in the associated samples.

Corrective Action: If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples. If the analyte that was present in the MB was not detected in the affected samples, then the data may be reported with qualifiers (check the project's requirements to be sure that this is allowed) and it must be addressed in the projects narrative.

9.3.3 Laboratory Control Sample (LCS)

9.3.3.1 With the exception of sample batches analyzed by Methods AK-102 or AK-103, one LCS is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The LCS consists of either 1 liter of organic-free water (for batches of aqueous samples) or 30 grams of Ottawa sand (or method designated sample size as

noted in Section 9.3.2 for batches of soil samples), to which the analytes of interest are added at a known concentration.

9.3.3.2 Methods AK-102 and AK-103 require the analysis of an LCS and a duplicate LCS (LCSD) with each preparation batch or every 20 samples, whichever is more frequent. The LCS and LCSD consists of either 1 liter of organic-free water (for batches of aqueous samples) or 30 grams (25 grams for AK102 and Ak103) of Ottawa sand (for batches of soil samples), to which the analytes of interest are added at a known concentration.

9.3.3.3 See Table III for spike levels and section 7.2.7 for preparation of the spiking solution. The LCS is processed exactly as samples in the batch and is used to assess the accuracy of the analytical system.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. For Alaska Methods AK102 and AK103, the acceptance limits are listed in Table V. Oklahoma method specifies a recovery of +/- 20% (80-120%) for waters and +/- 40% (60-140%) for soils. For all other methods, the control limits are set at ± 3 standard deviations around the calculated mean of the historical LCS recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

Corrective Action: If no MS was prepared for the batch or if the MS and MSD is analyzed and is out of control (per section 9.3.4 below) then the LCS must be assessed and must meet acceptance criteria. If LCS acceptance limits are not met, check the analytical system for proper function and calibration control, make any corrections, then the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the extraction process must be examined and corrected where problems are identified. The associated samples must then be re-extracted following the corrected procedure and reanalyzed. If the LCS recovery is above the upper control limit, and the associated samples are all below reportable concentrations, the deviation may be described in an NCM, if this is acceptable to the client or allowed by the specific program or project.

9.3.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

One matrix spike (MS) and one matrix spike duplicate (MSD) are prepared for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses real sample matrix in place of the blank matrix. Field blanks and equipment rinses may not be used to prepare the MS and MSD. The MS and MSD are processed exactly as samples in the batch, and are used to assess the effects of sample matrix on the accuracy and precision of the analytical system. See Table III for spike levels and see section 7.2.7 for preparation of the spiking solution.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. The control limits are set at ± 3 standard deviations around the calculated mean of the historical MS recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

The relative percent difference (RPD) between the MS and MSD must be less than the established control limit, which is based on 3 standard deviations of the mean of the historical data. RPD control limits are maintained in the laboratory LIMS.

Corrective Action: If the analyte recovery in the MS and/or the RPD between the MS and MSD fails acceptance criteria, but all other QC criteria are met, the MS/MSD failure may be attributed to matrix effects and the associated sample results may be reported as qualified. However, some programs (e.g., USACE) require reanalysis to confirm that presumed matrix effects are reproducible. The probable cause of the out of control event must be documented (significant concentration in parent sample, other matrix effects, non homogenous sample, etc.) in an NCM. Where no apparent cause is indicated then the batch should be evaluated for reextraction.

9.3.5 Surrogate Spikes

The calibration standards, field samples, and QC samples are spiked with ortho-terphenyl and n-octacosane surrogates. These surrogates have chemistry similar to the analytes of interest, but are not expected to be found in environmental samples. See Table III for spike levels and Sections 7.2.5 and 7.2.6 for surrogate spike preparation. Surrogate results are used to assess the performance of the analytical system for each field and QC sample.

Acceptance Criteria: The percent recovery of the surrogates must fall within the established control limits. For Alaska Methods AK102 and AK103, the acceptance limits are listed in Table V. For all other methods, the control limits are set at ± 3

standard deviations around the calculated mean of the historical surrogate recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are in control. Low surrogate recoveries in the blank require re-preparation and re-analysis of the samples, unless the sample surrogate recoveries are in control and the targeted compounds are not found in the affected samples.

If the sample surrogate recoveries are outside the established limits, verify calculations, dilutions, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference and the chromatogram should be examined for evidence of this. Low recoveries may be due to adsorption by the sample matrix (e.g., clay particles, peat, or organic material in the sample). Recalculate the results and/or reanalyze the extract if the checks reveal a problem.

If the surrogate recovery is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative. As with matrix spike failures, some programs (e.g., USACE) may require additional analyses to confirm suspected matrix interferences. The decision to reanalyze or flag the data should be made in consultation with the client. If matrix interference is not obvious, it is only necessary to re-prepare / reanalyze a sample once to demonstrate that a matrix effect is reproducible. The decision to re-prepare samples for out of control surrogate outside of the holding time should be made when consultation with the client indicates that this corrective action will be satisfactory.

9.3.6 RT Reference Standard

A combination of two n-alkane mixtures is analyzed to establish the retention time (RT) window for each initial calibration. The Tennessee / Mississippi DRO mixture is composed of all n-alkanes from C_{10} – C_{25} and the Calibration / Window Defining Hydrocarbon Standard is composed of all even-numbered n-alkanes from C_8 – C_{40}. The instrument conditions should be chosen so that each alkane component is completely resolved from the next alkane component (this is necessary in order to define each range as different from another specified range).

10.0 Calibration and Standardization

10.1 Instrumentation.

10.1.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.2 Instrument conditions are shown in Table IV.

10.1.3 The routine injection volume is 1 µL.

10.1.4 GC run conditions and columns must be chosen to meet the acceptance criteria for the RT Reference Standard listed in Section 9.3.6 and the calibration criteria in Section 10.

10.1.5 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder Arizona Calibration Training.

10.1.6 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist, GC and HPLC ICAL TALS Review Checklist. (See SOP DV-QA-0020.) Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.1.7 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and changing FID jets.

10.2 Calibration

10.2.1 The ICAL is performed using the concentration levels described in Sections 7.2.8 for diesel fuel, 7.2.9 for the jet fuels, and 7.2.10 for motor oil. Calibration levels are also presented in Table II. Although some methods allow as few as three calibration levels, the minimum number of calibration levels will be maintained as five levels for the purpose of this SOP.

10.2.1.1 Several separate initial calibration curves (ICALs) are required to calibrate for all the mixtures. An ICAL must always be maintained for the diesel fuel as these standards contain the surrogate compounds. ICALs for the other mixtures are analyzed as needed, depending upon the requested parameters. Samples may be calculated as one or more mixtures, dependent upon the project requirements.

10.2.1.2 The lowest calibration concentration is equal to the laboratory reporting limit (RL) concentration. The highest standard defines the highest sample extract concentration that may be reported without dilution.

10.2.1.3 It is not acceptable to remove points from a calibration curve for the purpose of meeting criteria, however, the lowest point or the highest point may be removed and the RL or maximum concentration

adjusted accordingly (as long as the minimum number of calibration points is sufficient for the curve used). Transfer calibration standard solutions into autosampler vials and load onto the GC autosampler. Use the Chem Station software to set up the analytical sequence.

10.2.2 The external standardization method is used. Tabulate the area response for each calibration level against the concentration injected. The ratio of the response to the concentration injected, defined as the calibration factor (CF), is calculated for the standard at each concentration as follows:

$$CF_i = \frac{A_{fuel}}{C_{fuel}}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.

A_{fuel} = Total area of the fuel calibration standard peak.

C_{fuel} = Concentration of fuel calibration standard, mg/mL

10.3 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through other options until the calibration criteria are met. Pay particular attention to the residuals noticed at the upper and lower end of the curves. This may be cause for rejection of a curve fit even if the calibration acceptance criteria are met.

10.3.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte or hydrocarbon range in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in section 10.2.2 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ *Equation 4*

Where:

CF_i = Calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\%$$ *Equation 5*

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}}$$ *Equation 6*

10.3.2 If the percent relative standard deviation (%RSD) for the average (mean) of the calculated calibration factors is less than 20%, the average calibration factor can be used for sample quantitation. Methods AK102 and AK103 require the % RSD to be <25%.

10.3.3 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered. SW-846 Method 8000B allows evaluation of the grand average RSD across all analytes, but TestAmerica-Denver evaluates each analyte individually.

Acceptance Criteria: The RSD must be ≤ 20% (AK102/AK103 upper limit is 25%).

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.3.4 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b$$ *Equation 7*

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b$$ *Equation 8*

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a}$$ *Equation 9*

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.3.5 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of a weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., AFCEE, DoD) require a correlation coefficient ≥ 0.995. Note that the AK102/AK103 method requires that r^2 be greater than or equal to 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.3.6 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 10}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.3.7 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Second-order regressions should be the last option. Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica Denver.

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH) of the expected concentration for that point. See the specific method for how to apply any exceptions (involves a narrowing of the concentration range to within the area that is within control).

10.3.8 Second-Source Initial Calibration Verification (ICV)

10.3.8.1 A second-source initial calibration verification (ICV) standard is prepared as described in section 7.2.14 and analyzed immediately after each ICAL. This standard can also be used as the continuing calibration verification (CCV) standard. The response for this standard must be within ± 20% of the response predicted from the ICAL (AK102/AK103 method requires the standard response to be within ± 25% and 8000B requires ± 15%).

10.3.8.2 The percent difference between the measured ICV calibration factor (or the measured concentration of the ICV standard) and the ICAL calibration factor (or the known concentration of the ICV standard) is calculated as follows:

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100\%$$

Where:

R_1 = Average calibration factor from the calibration curve or the ICV known value.

R_2 = Calculated calibration factor for the ICV analysis or the measured ICV value.

10.3.8.3 If the percent difference for the second-source verification falls outside of ± 20% (25% for AK102/AK103, ± 15% for 8000B), then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result fails, then re-prepare the verification standard, and/or re-prepare and

rerun the ICAL. The ICV must be analyzed under the same conditions that were used for the ICAL.

10.3.9 Continuing Calibration Verification (CCV)

10.3.9.1 A CCV standard (see Sections 7.2.11 through 7.2.13 for CCV standard concentrations) is analyzed at the beginning of the analytical sequence, every 12 hours of operation, or every 20 samples (whichever is more frequent), and at the end of the analytical sequence. A CCV must be included in each bracket for each fuel type that is requested and quantified for the samples and QC in the bracket.

10.3.9.2 The 8000 series methods (and some state specific methods-Arizona) indicate the need for two levels for the CCV where non-linear calibrations are used and all other methods indicate a single mid-point CCV. The response for this standard must be within ± 20% (25% for AK102/AK103) of the response predicted from the ICAL (see previous equation).

Note: it is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.3.9.3 If the percent difference between the measured CCV value and the expected CCV value falls outside of the method criteria, first check the accuracy of the CCV standard. Method criteria for %D for the CCV are:

- ± 20% for Method 8015C or 8015D (Method 8000C)
- ± 25% for AK102/AK103
- ± 15% for Method 8015B (8000B) and NWTPH

If the standard is accurate and the results fail acceptance criteria then the instrument must be recalibrated and all samples analyzed since the last successful CCV must be reanalyzed.

10.3.9.4 In some cases the nature of the samples being analyzed may be the cause of the failing %D. If such matrix effects are suspected then those samples must be reanalyzed (at a dilution if column damage is imminent) to prove matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

10.3.9.5 For any analyte not detected in the client samples, the %D for that analyte in the bracketing CCVs should also be within 20%, however, the results may be acceptable (with client approval) if the drift is positive (high). An NCM must be written to explain this case.

10.4 Retention Time Window Definition

10.4.1 Before establishing RT windows, be certain that the GC system is within optimum operating conditions. Analyze a diesel fuel CCV Standard (Section 7.2.11) and a Retention Time Reference Standard (section 7.2) three times each throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

10.4.2 Calculate the mean and standard deviation of the three absolute retention times for each carbon range of interest and each surrogate. Table 1 lists the boiling points for the aliphatic hydrocarbons used in the retention time reference standard.

10.4.2.1 The width of the retention time window for an individual peak is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period for each component.

10.4.2.2 In those cases where the calculated window for a particular analyte is less than the default window, use ± 0.05 minute (AK102 and AK103) +/- 0.03 (8000 methods) and +/- 0.1 min (Oklahoma) as the retention time window.

10.4.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor or appropriate second level review and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.1.1 Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. An NCM must be used for this documentation

11.2 Sample Preparation

Refer to extraction SOPs DV-OP-0006 for aqueous samples and DV-OP-0016 for soil samples. In the case of samples with an organic matrix, waste dilution SOP DV-OP-0012 may apply.

11.3 Instrument Maintenance

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance. If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control. The injector septum should be changed after about 200 injections have been completed. If the last CCV that was analyzed

indicated a high response then a simple liner change is typically sufficient to bring the system back into control. Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column. A reduced response may indicate that the system needs to be evaluated for leaks. Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed. Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.4 Analytical Sequence

11.4.1 The analytical sequence starts with a RT reference standard, an initial calibration (ICAL) and initial calibration verification (ICV) or with a continuing calibration verification (CCV) (see Section 10).

11.4.2 An instrument blank (injection of methylene chloride solvent) must be included in each analytical sequence. The instrument blank must meet the same acceptance criteria as the method blank (with the exception of surrogate recoveries).

11.4.3 A CCV standard is interspersed in the analytical sequence after 12 hours have elapsed or after 20 samples have been analyzed, whichever is more frequent. More frequent analysis of the CCV is recommended (and required by some programs) in order to minimize the number of samples needed to be reanalyzed in the event of an out of control recovery for the CCV.

11.4.4 Any sample suspected of being highly concentrated should be followed by an instrument blank to prevent carryover. If the blank analysis shows contamination beyond established “blank acceptance criteria”, the column must be baked out and subsequent blanks analyzed until the system is shown to be free from contaminants. If possible do not aliquot the entire sample volume. Use limited volume autosample vials so that an unused volume remains for possible repeat analyses.

11.4.5 If the measured concentration of any sample exceeds the highest calibration standard concentration, the sample extract must be diluted with methylene chloride and reanalyzed. See section 12.3 for dilution guidelines.

11.4.6 The analytical sequence is closed with a final CCV.

11.5 Daily Retention Time Windows

11.5.1 At the beginning of each daily analytical sequence, the RT Reference Standard (Section 7.2) is analyzed. The retention time windows for each diesel / motor oil range are adjusted based on the analysis of each n-alkane. The center of the retention time windows for the jet fuels are adjusted based on the analysis of the chromatography of any level of the jet fuel calibration standard. The center of the retention time windows must be updated at the beginning of each analytical sequence but not for any other CCV standards.

11.5.2 A notation is made in the run sequence log to identify any standard used for the retention time verification and adjustments.

11.6 Sample Analysis

11.6.1 Baseline Used for Integration

11.6.1.1 The same type of baseline must be applied equally to standards and samples. It is important that the baseline is drawn consistently by each analyst in the GC group, which must be part of the training for new analysts.

11.6.1.2 For the analysis of samples containing hydrocarbons that completely elute in the C_{10} to C_{28} range, the baseline should be drawn as shown in the example in Attachment 1. For routine analysis, this should be similar to the baseline produced by a method blank. Note that because of column bleed, this is not a flat, horizontal common baseline.

11.6.1.3 Measure the area of the methylene chloride blank projecting a horizontal baseline across the retention time range for DRO.

11.6.1.4 Valley-to-valley baselines should not be used. See the example in Attachment 2. Correct the instrument integration settings to minimize the number of manual integrations as shown in the example.

11.6.1.5 Samples containing high concentrations of hydrocarbons heavier than diesel do not completely elute in the C_{10}-C_{28} range. Precise and consistent integration can be difficult. Construction of the baseline requires some experience and judgment on the part of the analyst. Unless otherwise instructed in the client requirements, baseline to baseline integration will be used.

11.6.1.6 When such samples are encountered, it may be necessary to run an additional solvent blank to be sure that there is no carryover between samples.

11.6.1.7 The USACE requires construction of a baseline equivalent to that observed in the method blank (see the example in Attachment 3), unless approved project documents specify a different approach.

11.6.1.8 Some projects can require a common baseline from the signal at the beginning of the pattern to the signal at the end of the pattern (see the example in Attachment 4).

11.6.1.9 Always consult special project instructions for requirements for the baseline.

11.6.1.10 The requested fuel in the sample is determined by calculating the concentration of the target fuel using the summation of peak responses (i.e., peak areas) for all chromatographic peaks eluting between the retention time windows as determined in sections 10.4 and 11.4, using the average calibration factor or the calibration function determined in Section 10.

Using the average calibration factor, the concentration of the target fuel in the sample extract is calculated as follows:

$$C_e = \frac{A_e}{\overline{CF}}$$

Where:

C_e = Concentration of target fuel in the sample extract, mg/mL.

A_e = Total area of peaks for target fuel.

$\overline{CF}$ = Average calibration factor for target fuel.

11.6.2 Using the least-squares regression calibration, the concentration of the target fuel in the sample extract is calculated by solving the least-squares calibration equation for concentration as follows:

$$C_e = \frac{A_e - b}{m}$$

Where:

A_e = Area of the chromatographic peak for the target fuel in the sample extract.

m = Slope of the line as determined by the least-squares regression.

C_e = Concentration of the target fuel in the sample extract, mg/mL.

b = Intercept of the line as determined by the least-squares regression.

11.6.3 The concentration of the target fuel in the actual sample can then be calculated using the equation below. Actual calculations are performed by the chromatography software.

$$C = \frac{C_e V_e}{V_s} \times DF$$

Where:

C = Concentration of target fuel in original sample, mg/L or mg/kg.

C_e = Concentration of the extract that is injected into the GC. mg/mL.

V_e = Volume of the extract in mL.

V_s = Amount of sample extracted in L or kg.

DF = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, DF = 1 mL/mL.

If required for solid samples, the percent moisture calculation can be included in the data package. Percent moisture corrections are made automatically in the laboratory's LIMS.

11.6.4 Confirmation

Second column confirmation is not normally performed for this method as confirmation of chemical identity does not have to be specific.

11.6.5 Data Review

11.6.5.1 First level review is conducted by the analyst to ensure that all acceptance criteria are met, the analysis is properly documented, and the data are correctly uploaded into the LIMS. This is documented using the checklist prescribed in SOP DV-QA-0020, Organic Data Review.

11.6.5.2 Second level review is conducted by the supervisor of the group or an analyst appointed by the supervisor. Details of this review and the documentation are described in SOP DV-QA-0020. If problems are found with the data package, then the data are reviewed with the analyst.

11.6.5.3 Other reviews (PM Level III review and QA Data Reviews) are described in the TestAmerica Quality Assurance Manual (QAM), and associated TestAmerica policies and procedures.

12.0 Calculations / Data Reduction

12.1 Calculations

12.1.1 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.1.2 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA} \right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.1.3 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 17}$$

Where R_1 is the result for the MS and R_2 is the result for the MSD.

12.1.4 Concentration of Analyte in the Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.3 for details on establishing the calibration function):

Average Calibration Factor: $C_e = \frac{A_s}{\overline{CF}}$ *Equation 18*

Linear Regression: $C_e = \frac{\left[A_s - b\right]}{a}$ *Equation 19*

Non-Linear Regression: $C_e = f\left(A_s\right)$ *Equation 20*

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_s = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

$f(A_s)$ = Mathematical function established by the non-linear regression.

12.1.5 Concentration of Analyte in Original Sample (for 1 uL injection)

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \textit{Equation 21}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

12.2 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DEN-QA-0020.

12.3 Calibration Range and Sample Dilutions

12.3.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted with methylene chloride (record the lot number in the run sequence) and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e. the sample's concentration may be similar to or higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.3.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

13.1.1 An initial method detection limit study is performed in accordance with Policy DV-QA-005P. An MDL study is performed once a year to satisfy NELAC 2003 requirements. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly.

13.1.2 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are performed at least annually unless method requirements require a greater frequency.

13.2 Demonstration of Capabilities

13.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

13.2.2 IDOCs and on-going proficiency demonstrations are conducted as follows. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.2.3 The Oklahoma method requires that the analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

13.2.3.1 Analysis of 7 replicates of the diesel component standard at a concentration of 100 ug/L of total DRO in organic-free water with recoveries of all components within $\pm$ 40% of the known concentration and precision of all replicates within $\pm$ 30%.

13.2.3.2 Analysis of 7 replicates of DRO_free sand at a concentration of 10 mg/kg of total DRO with all recoveries within $\pm$ 40% of the known concentration and precision of all replicates within $\pm$ 30%.

13.2.3.3 As an exception use the NELAC recommended procedure for initial demonstration of capability in place of this procedure.

13.3 Training Requirements

The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention."

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- Vial waste – Expired Extract Vials – Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 Determinative Chromatographic Separations, , SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996

16.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.3 Method 8015B, Nonhalogenated organics by Gas Chromatography, Revision 2, December 1996.

16.1.4 Method 8015C, Nonhalogenated organics by Gas Chromatography, Revision 3, February 2007.

16.1.5 Method 8015D, Nonhalogenated organics by Gas Chromatography, Revision 4, June 2003.

16.1.6 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.7 Method 3546, Microwave Extraction, Revision 0, February 2007.

16.1.8 Method 3550B, Ultrasonic Extraction, Revision 3, December 1996.

16.1.9 Method 3550C, Ultrasonic Extraction, Revision 4, February 2007.

16.2 State of California "Leaking Underground Fuel Tank Field Manual", December 1987.

16.3 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

16.4 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

16.5 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

16.6 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified), Diesel Range Organics (DRO), October 22, 1997, Rev. 4.1.

17.0 Modifications from Source Methods

17.1 The California LUFT method uses carbon disulfide as the extraction solvent, whereas this SOP uses methylene chloride.

17.2 The California LUFT method uses a 20-gram sample size, which is extracted for four hours on a mechanical shaker. This SOP uses a 30-gram portion of sample, which is extracted per EPA Method 3550C (sonication).

17.3 Alaska Method AK103 for residual range organics (RRO) does not include analysis of aqueous samples. This SOP does provide for the determination of RRO, as defined in the Alaska method, in water.

17.4 The NELAC IDOC procedure is used in place of the Oklahoma IDOC procedure.

18.0 Attachments

Table I: Aliphatic Hydrocarbon Standard

Table II: Calibration Levels

Table III: Spike Levels for Quality Control

Table IV: Recommended GC Conditions

Table V: Alaska Methods AK102 and AK103 Acceptance Criteria for Quality Control

Table VI: DRO and RRO Method Summary Comparison Chart

Attachment 1: Routine DRO Integration

Attachment 2: Valley-to-Valley Integration Cannot Be Used

Attachment 3: Heavy Hydrocarbons with Baseline from Method Blank

Attachment 4: Heavy Hydrocarbons with Signal-to-Signal Integration

Attachment 5: Retention Time Reference Standard

Appendix 1: Oklahoma / Boeing Method Specifications

19.0 Revision History

- Revision 3, dated 04 April 2012
  - Updated section 6.2 with current supply list.
  - Added some current vendor sources in section 7.2 for materials used.
  - Added sections 7.3 Standards Verification, 7.4 Storage of Stock Standards, and section 7.5 Non-Routine Compounds.
  - Added holding time clarification to section 8.3.
  - Modified Section 10 for clarity and consistency with other SOP formats. Removed wording from section 10.3.4 that indicated the need for at least three multi-point curves in order to use a weighted regression. Changed the wording for section 10.4.2.2 to indicate when to use a default RT window.
  - Modified section 11 to include some guidelines for 11.3 Instrument Maintenance.
  - Modified section 12 to be consistent with other SOP formats.
  - Updated Table IV with current configurations.
  - Added Table VI and Attachment 5

- Revision 2, dated 25 March 2011
  - Updated SOP to include requirements for the various revisions of Methods 8000, 8015 and to include state methods for extractable hydrocarbons, including Alaska, California, Oklahoma and Washington.
  - Modified definitions for clarity
  - Updated section 13.

- Revision 1, dated 05 March 2010
  - Added section 6.3
  - Updated to match other GC SOP Format

- Revision 0, dated 31 January 2009

Table I: Aliphatic Hydrocarbon Standard

Compound Boiling Points		
n-Alkane	**Name**	**B.P.$_{760}$ (°C)**
C_8	Octane	126
C_{10}	Decane	174
C_{24}	Tetracosane	391
C_{28}	Octacosane	432
C_{32}	Dotriacontane	468
C_{36}	Hextriacontane	498

This Table can be used to get the estimated boiling point ranges of the hydrocarbons reported in a given sample.

Table II: Calibration Levels

Standard	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
DRO	0.1	0.25	1.00	5.00	7.50	15.00	30.00
Jet Fuels 4 and 8	0.1	0.5	1.00	2.50	5.00	10.00	NA
Motor Oil / RRO	0.1	0.25	1.00	5.00	7.50	15.00	30.0
Ortho-Terphenyl and n-octacosane (surrogates)	0.0005	0.00125	0.005	0.025	0.0375	0.075	---

(*All concentrations are in mg/L*.)

Table III: Spike Levels for Quality Control

Laboratory Control Samples (LCS) and Matrix Spike/ Spike Duplicate		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Soil (mg/kg)**
Diesel Range Organics	2.0	66.7
Jet Fuel 8	1.0	33.3
Jet Fuel 4	1.0	33.3
Residual Range Organics (or Motor Oil)	5.0	166.5

Surrogate Control Samples		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Low Soil (mg/kg)**
Ortho-Terphenyl	0.02	0.667
n-octacosane	0.02	0.667

Table IV: Recommended GC Conditions

Hydrogen Column Pressure	20 psi (U2) 15 psi (U)
Initial Column Temperature	45 ºC for 2.5 minutes (U2) 50 ºC for 2 minutes (U)
Temperature Ramp	25 ºC / minute (U2) 35 ºC / minute(U)
Final Column Temperature	325 ºC for 3.3 minutes (U2) 330 ºC for 5 minutes (U)
Injector Temperature	285 ºC
FID Temperature	325 ºC

Table V. Alaska Methods AK102 and AK103 Acceptance Criteria for Quality Control

Method AK102 (DRO) Acceptance Criteria				
QC Parameter	**Water (mg/L)[1]**	**Soils (mg/kg)[1]**	**% Recovery**	**RPD (%)**
Lab Fortified Blanks (LCS)	0.5 – 2.0	---	75 - 125	20
Continuing Calibration (Includes surrogate compounds)[2]	---	---	75 - 125	---
Calibration Verification	---	---	75 - 125	---
Surrogate Recovery for "Laboratory Control Sample"[2]	0.02	0.8	60 - 120	---
Surrogate Recovery for Field Samples	0.02	0.8	50 - 150	---
Method AK103 (RRO) Acceptance Criteria				
QC Parameter	**Water (mg/L)[1]**	**Soils (mg/kg)[1]**	**% Recovery**	**RPD (%)**
Lab Fortified Blanks (LCS)	---	500	60 - 120	20
Continuing Calibration	2000	---	75 - 125	---
Calibration Verification	2000	---	75 - 125	---
Surrogate (n-triacontane-d_{62}) Recovery for Control Samples[2]	---	50	60 - 120	---
Surrogate (n-triacontane-d_{62}) Recovery for Field Samples	---	50	50 - 150	---

NOTE: The information in this table is taken verbatim from the referenced Alaska methods. This SOP provides for spiking both DRO and RRO in water and soils. The acceptance limits in this table are used when client or project requirements specify compliance with the Alaska methods.

---

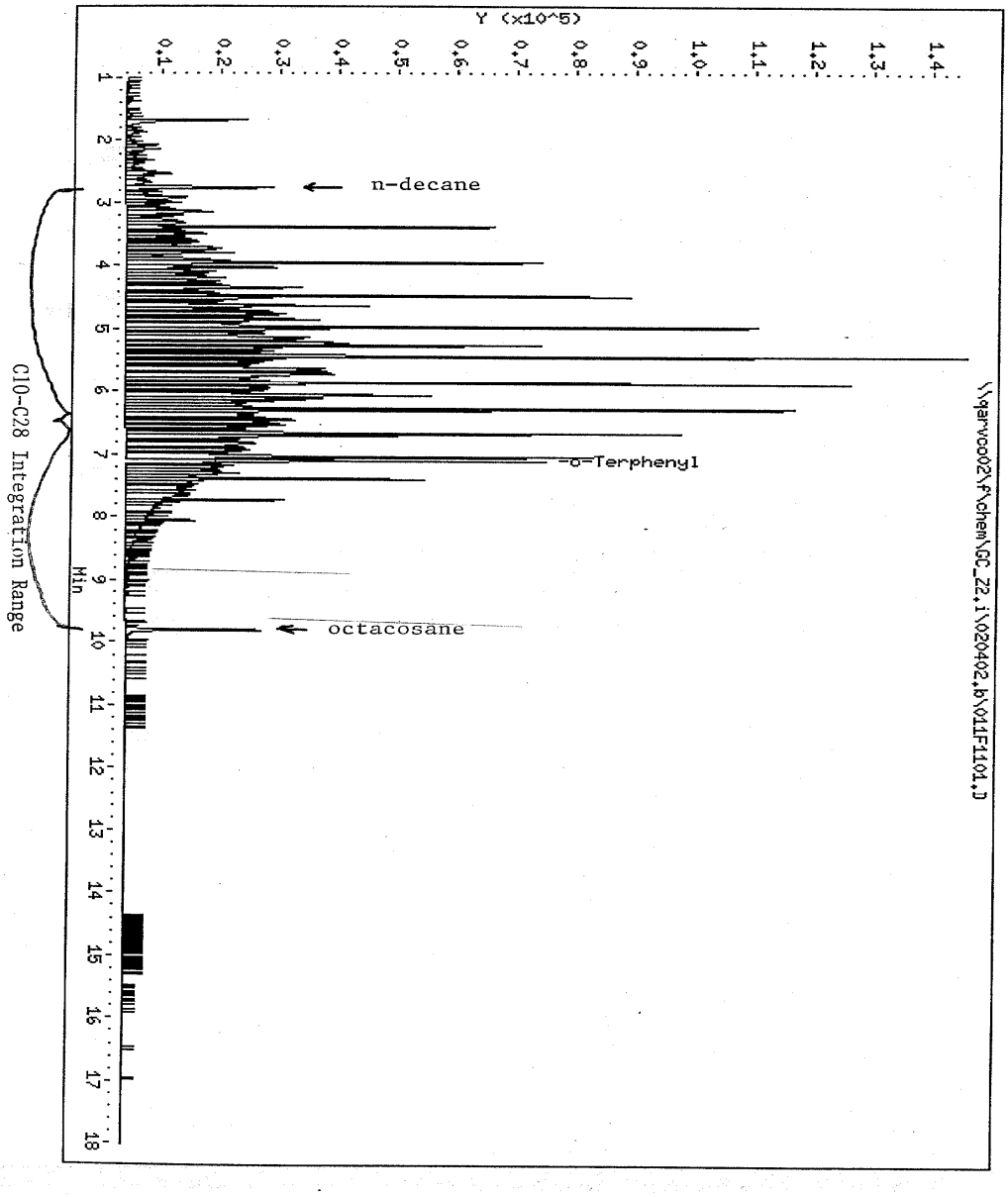
1 Suggested concentrations. May vary with matrix.

2 According to the Alaska methods, this is any laboratory prepared samples used for quality control, except calibration standards. At TestAmerica Denver, this includes the LCS, method blank, MS, and MSD. The continuing calibration for surrogates is verified at the instrument by including them in the continuing calibration DRO sample.

Table VI. DRO and RRO Method Summary Comparison Chart

Program	Standards Surrogates	Ranges Min RT Windows	Batch Size	MB RL	ICAL # Points RSD r	CCV Bracket size	LCS/MS/Other Sample Size
8015B	Commercial Diesel	C_{10} - C_{28} Apex ± window	20 Samples	Blank subtraction is allowed	5 levels; lowest at RL % RSD < 20	CCV/12 hour window & close; mid range ± 15% All compounds in RT window	Standard QC model
8015C	Commercial #2 Diesel 6 month expiration Surrogate: o-terphenyl	C_{10} - C_{28} Apex ± window	20 Samples	Blank subtraction is allowed	5 levels; lowest at RL % RSD < 20	CCV/12 hour window & close; mid range ± 20% All compounds in RT window	Standard QC model
8015D	Commercial #2 Diesel 6 month expiration	C_{10} - C_{28} Apex ± window	20 Samples	Blank subtraction is allowed	5 levels; lowest at RL % RSD < 20	CCV/12 hour window & close; mid range ± 20% All compounds in RT window	Standard QC model
8000B	N/A	Default 0.03min; ± 3 standard deviation over 72 hour period; Use center of CCV in beginning or mid-point standard of ICAL *New RT windows with new column instillation.	20 Samples	Less than RL Less than 5% of sample result	5 Levels. Lowest ≤ RL % RSD < 20% (mean RSD allowed). r ≥ 0.99 6 level for quadratic COD> 0.99	CCV/12 hour window & close; mid range: every 10 samples recommended ± 15%D (grand mean allowed) 2 levels for quadratic curve	In house limits
8000C	N/A	Default 0.03min; ± 3 standard deviation over 72 hour period; Use center of CCV in beginning or mid-point standard of ICAL *New RT windows with new column instillation.	20 Samples	Less than RL	% <20; r ≥ 0.99 5 levels; r² > 0.99 Batch calculation is suggested (20% D)	± 20% 2 different concentrationsfor quadratic curves	In house limits
DRO/AK102	Commercial #2 Diesel o-terphenyl (60-120% - LCS) (50-150% - Samples)	Beginning C_{10} to beginning C_{25} ± 0.05 min	20 Samples	20 mg/Kg 800 ug/mL Less than PQL	r² > 0.995 At least 3 standards % RSD <25%	ICV ± 25% CCS/CCV ± 25% Every 20 samples Midpoint of the curve	
RRO/AK103	Equal weights of 30W and 40W motor oil. Surrogate: n-Triacontone	Beginning C_{10} to end C_{36} ± 0.05 min	20 Samples	100 mg/Kg	r² > 0.995 At least 3 standards % RSD <25%	ICV ± 25% CCS/CCV ± 25% Every 20 samples Midpoint of the curve	RL - X5 MDL LFB = 60-120%
Oklahoma	Ten component blend of alkanes; 10-28 even (store freezer); 6 months expiration	0.1 min before C_{10} to 0.1 min after C_{28} (0.1 min)	20 Samples; Duplicate spikes evrey 10 samples	0.1 mg/L 10 mg/Kg Less than PQL	minimum 3 levels Lowest at PQL r at least 0.99	CCV ± 20% daily Has special IDOC (see method)	Sample size discresionary; at least 800 mL. Soil not to exceed 20 g. Concentrate to 1 mL LCS/LCSD & MS/MSD - ± 20% water:± 40% soil
NWTPH	#2 Diesel or known other component. Surrogate: o-terphenyl	Must incorporate most of components; break where multi components occur	MB per 20 samples; Duplicate per 10 samples	Diesel: 25 mg/Kg; 0.25 mg/L Motor Oil: 100 mg/Kg; 0.5 mg/L	5 points r > 0.990 Back quantitate standards to ± 15%	± 15% midpoint opening and closing	20 g soil 400 mL water
TX1005	Unleaded Gasoline and #2 Diesel	RF for C_{35} ≥ 75% of the RF of C_{28} Beginning 0.1 min after the RT of 1st marker compound and ending 0.1 after the RT of the ending marker compound. Two ranges; C_6 - C_{12}; >C_{12} - C_{28} default of ± 0.1 minute	20 Samples	Less than RL	5 point calibration Peak to valley integration for C_6 - C_{12} Forced baseline projection for >C_{12} - C_{28} r²≥ 0.995 At least 3 standards % RSD <25%	Mid-point cal standard performed at the beginning and end of each batch, shift, or work day, whichever is more frequent. RPD ± 25%	%R within 70-130% and RPD ± 20% or within established control limits

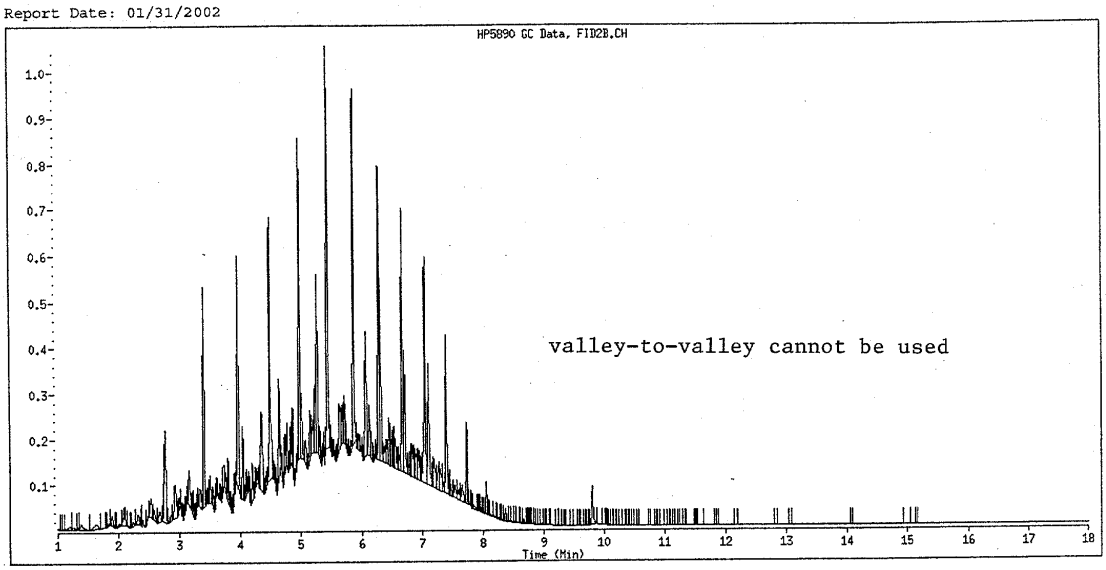
Attachment 1
Routine DRO Integration



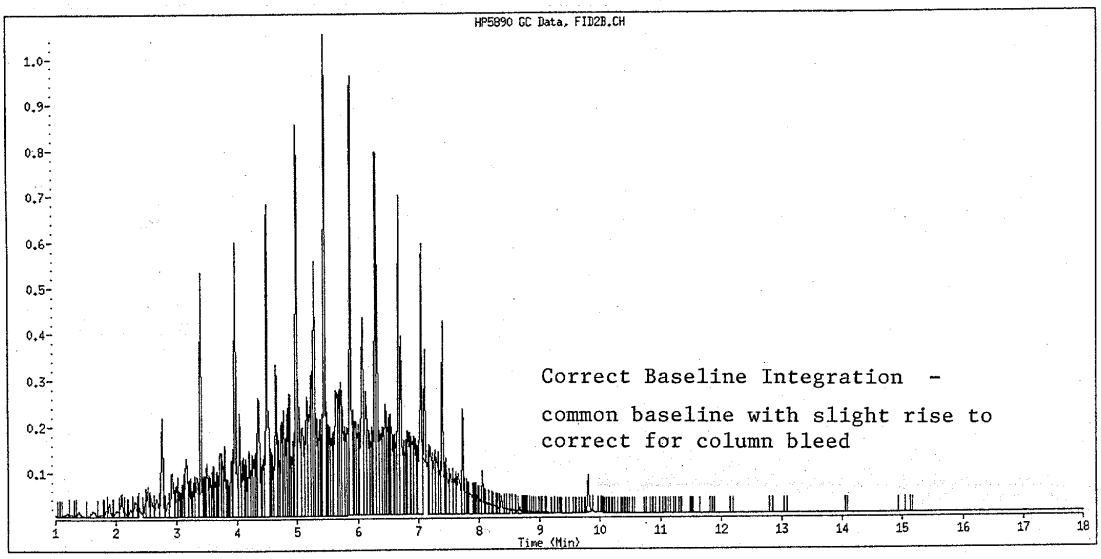
Attachment 2
Valley to Valley Integration cannot be used

MANUAL INTEGRATION TO PRODUCE CORRECT BASELINE

Report Date: 01/31/2002



Original Integration

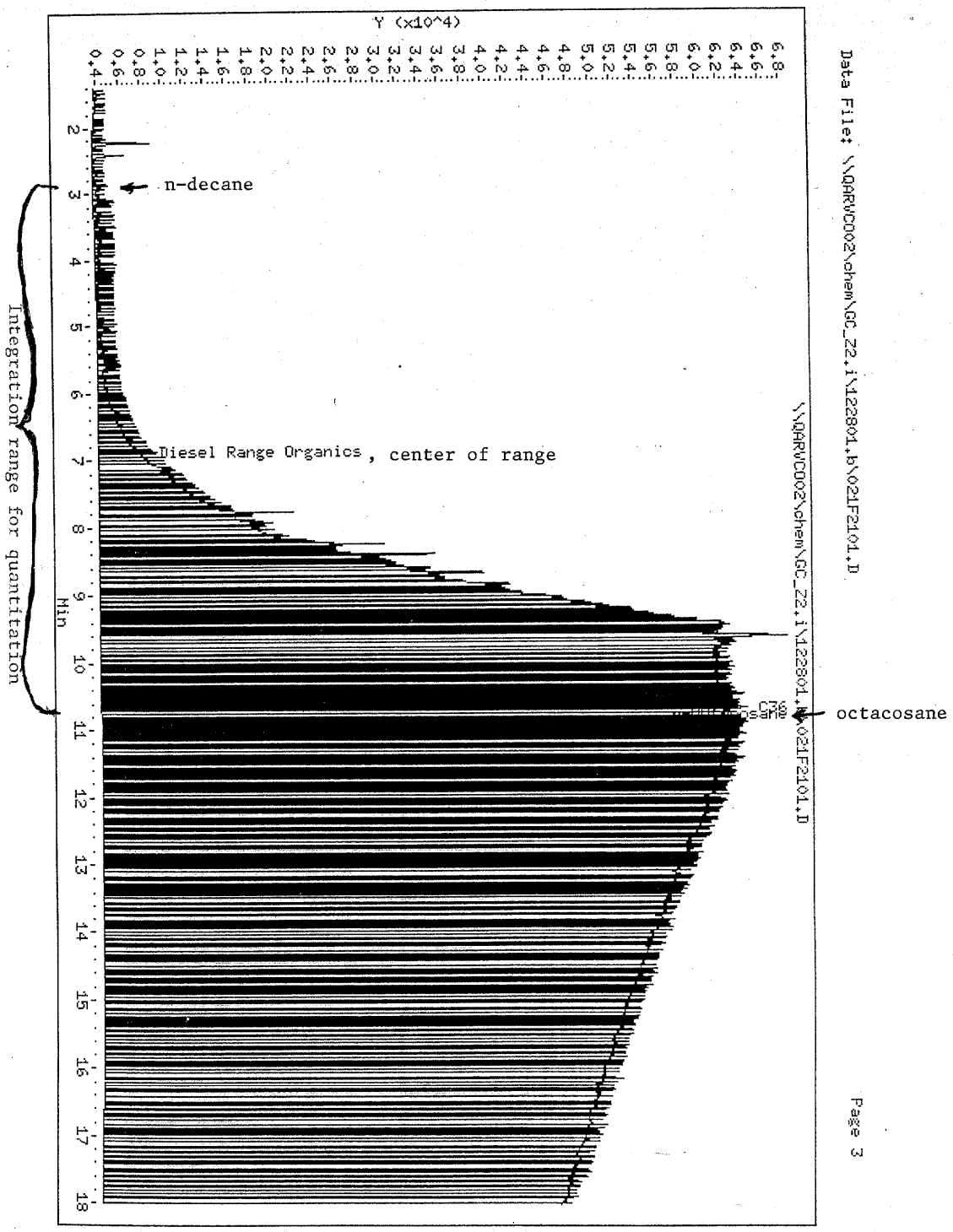


Manual Integration

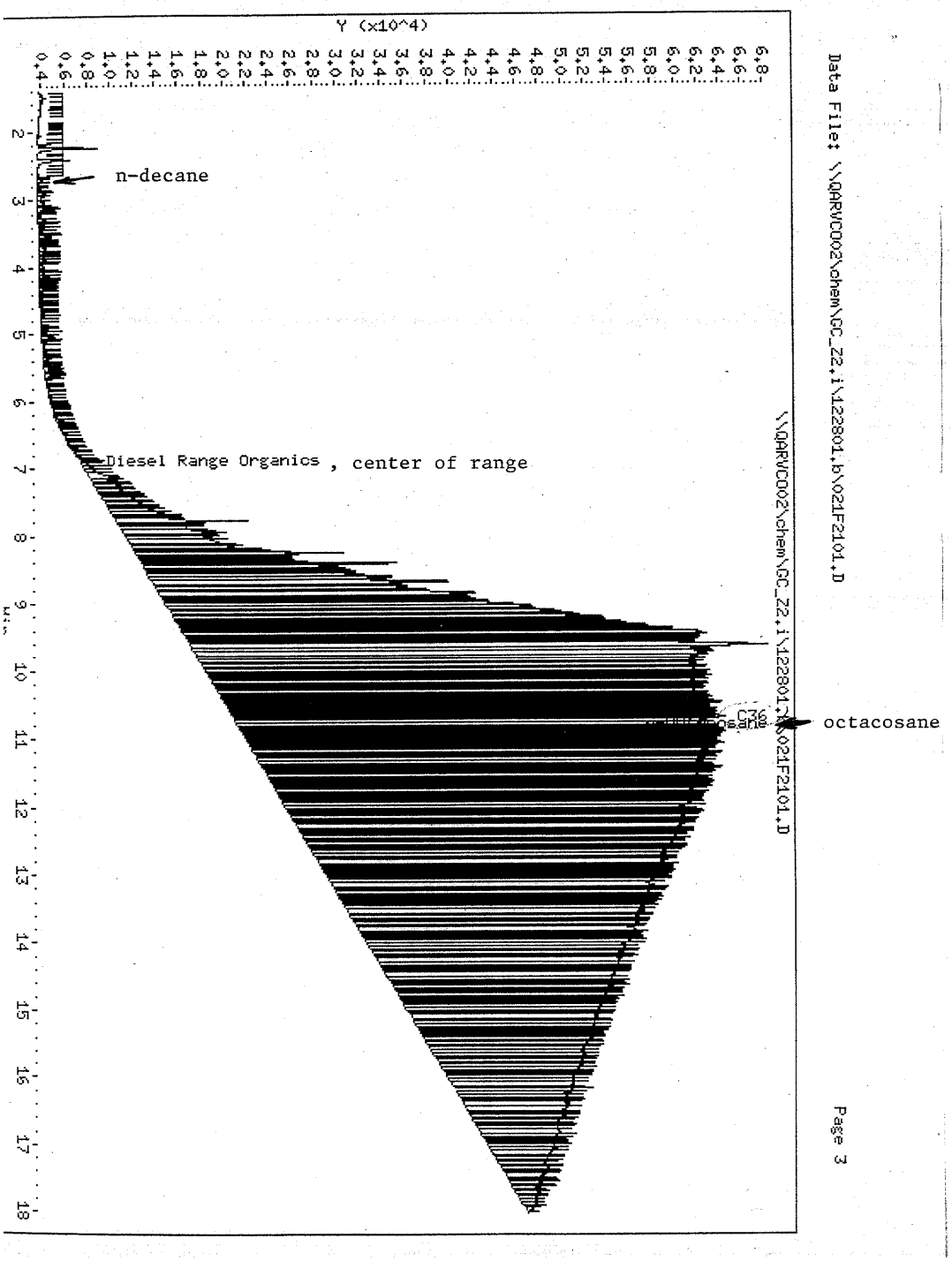
Manually Integrated By: fiedlerh
Manual Integration Reason: Baseline Event

1/31/12

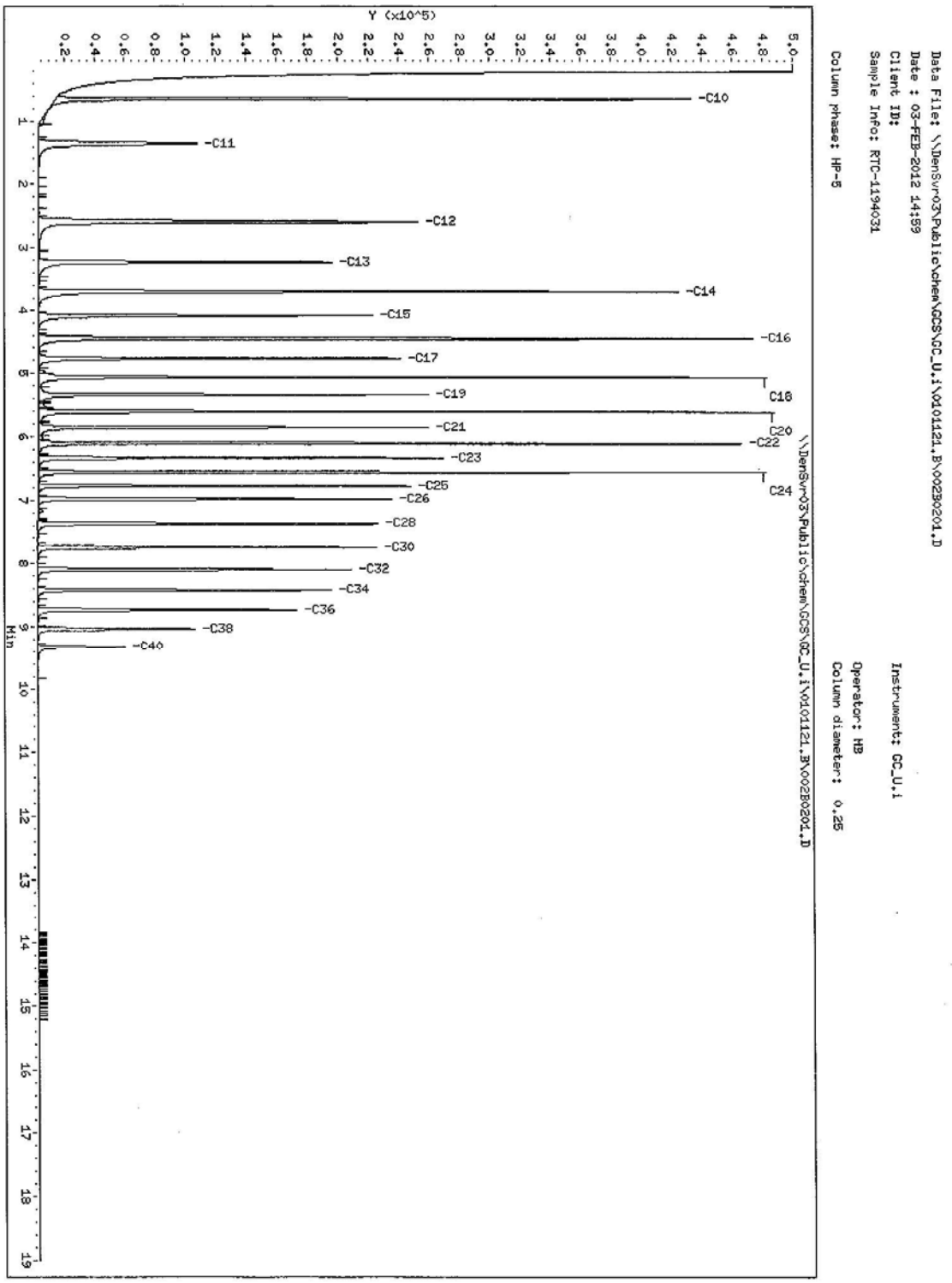
Attachment 3
Heavy Hydrocarbons with Baseline from Method Blank



Attachment 4
Heavy Hydrocarbons with Signal to Signal Integration



Attachment 5
Retention Time Reference Standard



Appendix 1: Oklahoma / Boeing Specifications

Definitions

C_8-C_{11}; C_{12}-C_{14}; C_{15}-C_{20}; C_{21}-C_{30}. Each of these carbon ranges are determined by the retention time defining standard. In terms of retention time, the lab will include instrument response eluting 0.1 minutes prior to the apex of the peak for the first alkane of a given carbon range. The end of a given carbon range is defined by the retention time for the alkane marker that designates the beginning of the next carbon range, finally ending 0.1 minutes after the elution of the C_{30} marker. Identification and quantification of TPH components requires more analytical judgment than other GC methods. The TPH chromatograms consist of groups of peaks that fall within a noted carbon retention time range. Diesel Fuel will be defined as C_8-C_{30} for the Oklahoma method.

The standard used for this calibration is a mixture of the even-numbered n-alkanes from C_{10} – C_{28}. To calibrate for this method, the following determinations are used:

C_8 – C_{11} range: based on the response of C_{10}
C_{12} – C_{14} range: based on the responses of C_{12} and C_{14}
C_{15} – C_{20} range: based on the responses of C_{16}, C_{18} and C_{20}
C_{21} – C_{30} range: based on the responses of C_{22}, C_{24}, C_{26}, and C_{28}

Standards:

Stock Oklahoma Standard: Supelco TPH1 Standard at 2000 μg/mL of each n-alkane compound.

Stock Oklahoma Second Source Standard: Ultra Scientific EPA/Wisconsin Standard at 2000 μg/mL of each n-alkane compound.

Stock Oklahoma Surrogate Standard: Supelco o-terphenyl solution at 10,000 μg/mL in methylene chloride.

Oklahoma Surrogate Spike Solution: Dilute 5 mLs of Stock Oklahoma Surrogate Solution to a final volume of 200 mL using 90:10 acetone:methylene chloride for a final concentration of 50 μg/mL.

Oklahoma Spike Solution: Dilute 12.5 mLs of Stock Diesel Fuel Second Source Standard to a final volume of 50 mL using acetone for a final concentration of 500 μg/mL of each n-alkane.

Appendix 1: Oklahoma / Boeing Specifications (continued)

Oklahoma / Boeing Calibration Standards: Diesel fuel standards for the Boeing and Oklahoma methods are prepared in methylene chloride at 6 concentration levels using the Stock Oklahoma Standard and the Stock Oklahoma Surrogate Standard as defined for this method.

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc of each n-alkane (μg/mL)	Surrogate Conc (μg/mL)
6	Stock + Stock Surrogate	0.75 .15	1.0	1500	1500
5	Level 6	.2	0.6	500	500
4*	Stock + Stock Surrogate	0.25 0.05	5	100	100
3	Level 4	0.5	1.0	50	50
2	Level 4	0.25	1.0	25	25
1	Level 4	0.1	1.0	10	10

NOTE: The Level 4 (*) calibration standard is also used as the continuing calibration verification standard.

Oklahoma Second Source: The second source for this method is prepared by diluting 0.05 mL of the Stock Oklahoma Second Source Standard and 0.01 mL of the Stock Oklahoma Surrogate Standard to a final volume of 1 mL.

Calibration Levels:

Standard	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
$C_8 – C_{11}$	10	25	50	100	500	1500
$C_{12} – C_{14}$	20	50	100	200	1000	3000
$C_{15} – C_{20}$	30	75	150	300	1500	4500
$C_{21} – C_{30}$	40	100	200	400	2000	6000
$C_8 – C_{30}$	100	250	500	1000	5000	15000
Ortho-Terphenyl (surrogate)	10	25	50	100	500	1500

(*All concentrations are in mg/L*.)

Appendix 1: Oklahoma / Boeing Specifications (continued)

Spike Levels for Quality Control

Laboratory Control Samples (LCS) and Matrix Spike/ Spike Duplicate		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Soil (mg/kg)**
$C_8 – C_{11}$	0.5	10
$C_{12} – C_{14}$	1.0	20
$C_{15} – C_{20}$	1.5	30
$C_{21} – C_{30}$	2.0	40
$C_8 – C_{30}$	5.0	100

Surrogate Control Samples		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Low Soil (mg/kg)**
Ortho-Terphenyl	0.05	1

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Title: Gasoline Range Organics (GRO) by GC/FID [SW846 Method 8015 and others]

Approvals (Signature/Date):

Dennis Jonsrud — 7-14-11 — Date
Technical Manager

Adam Alban — 19 July 11 — Date
Health & Safety Manager / Coordinator

John Morris — 7/25/11 — Date
Quality Assurance Manager

Robert C. Hanisch — 7/20/11 — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

GRO components correspond to an alkane range from n-hexane (C_6) to n-decane (C_{10}), with boiling points between approximately 60 °C and 170 °C. The method is sensitive to a variety of aromatic and aliphatic hydrocarbons that can be present in a wide range of petroleum distillates and petroleum products (e.g., automotive gasoline, jet fuels, lighter fluid, and some paint strippers).

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is used to determine the concentration of gasoline range organic (GRO) materials in water and soil. This procedure is based on EPA Method 8015B, 8015C, 8015D, and associated methods 8000B and 8000C. Also represented are Alaska Method AK101, Washington method NWTPH-Gx and Oklahoma Methods 8020/8015 (modified Gasoline Range Organics.

1.1.2 The standard laboratory reporting limit (RL) for this method is 1.2 mg/kg for soils and 25 μg/L for water for all methods, both soil and water except for the Oklahoma methods in water where the standard RL is 10 ug/L. The method RL indicated by NWTPH-Gx is 5 mg/kg for soil and 250 ug/L for water, Alaska AK101 20 mg/kg soil and 100 ug/L water and in each case the standard RL is lower than the method specific RL. For Oklahoma the method RL is stated as 0.1 mg/kg for soil and 20 ug/L water. The higher laboratory soil RL for the Oklahoma method is due to the fact that the laboratory MDL does not support this method's lower soil RL.

1.1.3 Unless otherwise noted the most stringent or common criteria is reported and adhered to for the methods listed. Consistent with SW-846, method references will not reference a specific version unless it is necessary to state criteria for that version.

1.1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 19 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Water samples are analyzed directly for gasoline range organics using the SW-846 Method 5030 purge-and-trap extraction and Method 8015 GC analysis. An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto the gas chromatograph column for analysis.

2.2 Solid samples are preserved in and extracted with methanol by the SW-846 Method 5035 high-level option. An aliquot of the methanol extract is added to organic-free reagent water, which is then analyzed using the SW-846 Method 5030 purge-and-trap extraction and Method 8015 GC analysis. The Alaska AK101 method requires

dispersion of soil and waste samples in methanol that contains the method surrogates at the time of sampling in the field to dissolve and preserve the volatile organic constituents.

2.3 Method 8015 is a gas chromatography (GC) method. Detection is achieved by a flame ionization detector (FID), which can be used with a photoionization detector (PID) in series to characterize individual aromatic components by method 8021B (see SOP DV-GC-0023 for details). The GRO retention time range is characterized by running n-alkane standards. Quantitation of GRO organics is based on the FID detector response to an external gasoline standard relative to an internal standard.

3.0 Definitions

3.1 Gasoline Range Organics (GRO): All chromatographic peaks eluting between 2-methylpentane and 1,2,4-trimethylbenzene are attributed to GRO. Quantitation is based on a direct comparison of the area within this range to the total area of the calibration standard within this range.

3.2 Alaska Method AK101 defines GRO as all chromatographic peaks, both resolved and unresolved, eluting between the peak start time for C_6 (n-hexane) and the peak start time for C_{10} (n-decane). Quantitation is based on a direct comparison of the baseline-to-baseline integrated area within this range to the total area of the calibration standard over the same (C_6 – C_{10}) range, using FID response.

3.3 Oklahoma Methods 8020/8015 (modified) defines GRO as all chromatographic peaks, both resolved and unresolved, eluting 0.1 minutes before the peak start time for MTBE and 0.1 minutes after the peak start time for naphthalene.

3.4 Method NWTPH-Gx requires the retention time range (window) for gasoline integration to include, at a minimum, toluene through naphthalene.

3.4.1 Surrogate peak areas must be determined by valley-to-valley integration.

3.4.2 Other volatile petroleum products may be analyzed by method NWTPH-Gx provided that they elute within the gasoline range and the window is adjusted to encompass the expected range of the product and co-eluting surrogate areas are subtracted.

4.0 Interferences

4.1 High levels of heavier petroleum products such as diesel fuel may contain some volatile components producing a response within the retention time range for GRO. Other organic compounds, including chlorinated solvents, ketones, and ethers, are also detectable by this method. As defined in the method, the GRO results will include these compounds.

4.2 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank prepared from reagent water (for water samples) or methanol (for soil and sediment samples that will be preserved with methanol in the field) and carried through sampling and subsequent storage and handling can serve as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and purging device must be replaced or thoroughly rinsed between samples with methanol and then dried at 105°C in an oven. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of an instrument blank to check for cross contamination. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds, or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, rinse with methanol, and then dry in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bake-out and purging of the entire system may be required. Screening of all samples prior to analysis is recommended to protect analytical instrumentation.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view. There should also be a warning label/sticker on each instrument that identifies it as a UV light source.

5.1.4 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.5 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Gas chromatographic analytical system complete with gas chromatograph suitable for purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system capable of determining peak areas is recommended.

6.1.2 Columns

6.1.2.1 Restek Rtx® 502.2, 105 m, 0.53 mm I.D., 0.3 micron film thickness, or equivalent.

6.1.2.2 For 8021B/GRO and the Oklahoma Methods 8020/8015 (modified), Restek Rtx-1 ®, 105 m, 0.53 mm I.D., 3 micron film thickness or equivalent.

6.1.2.3 Other capillary columns such as a 30 m, 0.53 mm ID, DB-5 may be used, but the column must be able to resolve 2-methylpentane from the methanol solvent front in a 30 μg/L LCS standard and should resolve ethylbenzene from m/p-xylene.

6.1.3 Detectors: Flame ionization detector (FID) in series with a photoionization detector (PID). The FID is used for the measurement of all hydrocarbons. The optional PID is to be used only for the measurement of volatile aromatics for 8021B/GRO and the Oklahoma Methods 8020/8015 (modified).

6.1.4 Purge-and-Trap Device: Tekmar LSC 2000 with optional ALS 2016 autosampler or equivalent for Method 5030 preparation.

6.1.5 An analytical balance capable of accurately weighing 0.0001 g is used for preparing standards. A top-loading balance capable of weighing to the nearest 0.01 g is used for weighing samples. The accuracy of each balance is checked each day of use (see SOP DV-QA-0014 for details).

6.2 Supplies

6.2.1 Syringes: 5 mL Luerlock glass hypodermic and a 5 mL gas-tight syringe with shutoff valve.

6.2.2 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 500 mL, and 1,000 mL with a ground-glass stopper.

6.2.3 Microsyringes: 1 µL, 5 µL, 10 µL, 25 µL, 100 µL, 250 µL, 500 µL, and 1000 µL.

6.2.4 Syringe Valve: Two-way, with Luer ends (three each), if applicable to the purging device.

6.2.5 Glass Scintillation Vials: 20 mL, with screw-caps/crimp caps and Teflon liners or glass culture tubes with a screw-cap and Teflon liner, or equivalent.

6.2.6 Stainless steel spatula.

6.2.7 Wooden tongue depressors.

6.2.8 Disposable Pasteur pipettes

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Reagent Water: De-ionized reagent water that is boiled and purged with helium.

7.1.2 Methanol: Purge and trap grade or equivalent. Store away from other solvents. See SOP CA-Q-S-001 for the description of the program for testing solvents prior to use.

7.1.3 Methanol: HPLC grade, used for cleaning labware.

7.1.4 Sand: Reagent grade Ottawa Sand or equivalent, dried in an oven at 105 °C for 8 hours before using.

7.2 Gasoline Calibration Standards

NOTE: Specifics for preparing diluted standard solutions depend on the concentration of the original stock standard. The source of the stock or parent neat gasoline standard depends on the specific client or project requirements and can change from project to project. The details presented in this section are intended to provide guidance only. Specific dilutions may change depending on the source of the gasoline standard required for the project.

7.2.1 Gasoline Standards Sources

Authentic gasoline fuel standards are used for calibrations and spiking. Depending on project/client requirements, the gasoline standard may be any of the following:

7.2.1.1 A neat (100%) gasoline obtained from a gasoline station, either locally or from the sampling region, as directed by client requirements. NWTPH-Gx calls for the use of unleaded regular gasoline.

7.2.1.2 When analyzing for both Method 8015B GRO and Method 8021B BTEX, a certified mix of gasoline and aromatic hydrocarbons (BTEX) is used. See Table 1.

7.2.1.3 For Alaska Method AK101, an equal-weight mixture of regular, plus, and premium grades of commercial, non-oxygenate gasoline is used. This standard may be obtained as a neat gasoline or as a concentrated stock standard solution in methanol from a commercial source. See Table 2.

7.2.2 Preparing GRO Standards from Neat Gasoline

7.2.2.1 To prepare a stock standard from neat gasoline, first determine the density of the neat standard by weighing 2 µL of the standard in a tared 10 µL syringe. The density is calculated as follows:

$$\rho = \frac{m}{v} \qquad \textit{Equation 1}$$

Where:

ρ	=	Density in g/µL
m	=	Mass in grams
v	=	Volume in µL

7.2.2.2 Preparation of GRO Stock Standard, 10,025 µg/mL

To prepare a stock standard from neat gasoline, dilute a volume of the neat gasoline, as determined based on its density, to 5 mL with

methanol to achieve a final concentration of 10,025 µg/mL. The volume of the neat gasoline needed is determined as follows:

$$\text{Volume of Neat Gasoline}\,(\mu L) = \frac{10025(\mu g/mL)\times 5(mL)}{\rho(g/\mu L)\times 1000(\mu g/g)} \qquad \textit{Equation 2}$$

This calculation will generally yield a result somewhat greater than 50 µL.

7.2.3 Commercial Calibration Stock Standard, 10,025 µg/mL

As described in Section 7.2.2.2, a GRO stock standard may be prepared using a neat gasoline standard. Alternatively, a stock GRO or combined GRO and aromatic solvent (BTEX) stock standard may be purchased from a commercial vendor with a GRO concentration of 10,025 µg/mL in methanol. See Tables 1 - 3.

7.2.4 Calibration Intermediate Standard, 200.5 µg/mL

Dilute 36 µL of the stock standard (Section 7.2.2.2 or 7.2.3) with 1.8 mL of methanol to achieve a final GRO concentration of 200.5 µg/mL. See Table 1. (Other stock standard concentrations will require different aliquots to prepare the intermediate standard.)

7.2.5 Working Level Calibration Standards

7.2.5.1 Method 8015 GRO/AK101 Analysis

The initial calibration is performed at 6 concentration levels for the Method 8015 GRO/AK101 analyses as shown in the table below and in Table 4. Each calibration level standard is prepared at the instrument just prior to analysis and is analyzed in the same manner as an aqueous sample (see Section 10.7). Use the following table to inject the appropriate amount of the calibration intermediate standard solution (Section 7.2.4) and the surrogate working solution (Section 7.3.2) into the 5 mL Luerlock syringe containing 5 mL of reagent water. Other calibration standards can be made up based on concentrations of the intermediate stock standards and components required by the program or client.

Preparation of Working Level Calibration Standards for GRO Analyses

Level	Vol. of Intermediate & Surrogate Working Standards (µL)	Final Vol (mL)	Final GRO Concentration (µg/L)	Final Surrogate Concentration (µg/L)
1	0.5	5.0	20	3
2	1.25	5.0	50	7.5
3	2.5	5.0	100	15
4	5	5.0	200	30
5	12.5	5.0	500	75
6	25	5.0	1,000	150

7.2.5.2 For soil samples the same curve is used and the concentrations are

recalculated based on the default sample size used.

7.2.6 Second-Source Initial Calibration Verification (ICV) and Spike Standard

NOTE: When analyzing aqueous samples, the second-source ICV working standard solution is used to prepare the laboratory control sample (LCS), which then serves the dual purpose of LCS and ICV. When analyzing soil samples, the LCS is prepared by spiking reagent sand with the ICV stock solution and extracting into methanol.

Obtain a neat gasoline standard or commercially available gasoline mix standard from a source different than the source that supplied the primary standard (Section 7.2.5) and prepare according to section 7.2.2.7 and 7.2.2.8.

7.2.6.1 ICV/Spike Stock Standard Solution, 5500 µg/mL

If using a neat gasoline, prepare an ICV/spike stock standard solution at a concentration of 5500 µg/mL. Determine the density of the neat standard as described in Section 7.2.2. Calculate the volume of the neat standard needed to prepare 5 mL of stock standard using the following equation:

$$\text{Volume of Neat Gasoline}\,(\mu L) = \frac{5500(\mu g/mL)\times 5(mL)}{\rho(g/\mu L)\times 1000(\mu g/g)}$$ *Equation 3*

Dilute the calculated volume of neat standard with methanol to a final volume of 5 mL.

Alternatively, obtain a stock GRO standard solution in methanol from a commercial source and dilute as necessary with methanol to achieve a final concentration of 5500 µg/mL. See Table 8.

7.2.6.2 ICV/Spike Working Standard Solution, 100.8 µg/mL

Prepare an ICV/spike working standard solution at a concentration of 100.8 µg/mL by diluting 33 µL of the ICV/spike stock standard (Section 7.2.6.1) with methanol to a final volume of 1.8 mL. See Table 8.

7.2.7 Continuing Calibration Verification (CCV) Standard, 200 µg/L

7.2.7.1 The CCV for Method 8015 GRO/AK101 analyses is prepared in the same manner as the Level 4 working level calibration standard (Section 7.2.5), which has a concentration of 200 µg/L, as the CCV standard.

7.2.7.2 The CCV for Method 8021B/GRO is prepared in the same manner as the Level 5 working level calibration standard (Section 7.2.5) which has a concentration of 240 µg/L, as the CCV standard.

7.2.7.3 The CCV for the Oklahoma Methods 8020/8015 (modified) is prepared in the same manner as the Level 5 working level calibration

standard (Section 7.2.5) which has a concentration of 500 μg/L, as the CCV standard.

7.3 Surrogate Standards

7.3.1 Surrogate Stock Standard, 2,000 μg/mL

7.3.1.1 The surrogate used for this method is α,α,α-trifluorotoluene. A stock solution is obtained from a commercial vendor at a concentration of 2,000 μg/mL in methanol.

7.3.1.2 For AFCEE projects or as specified, chlorobenzene can be used in place of α,α,α-trifluorotoluene, using the same concentrations. A stock solution of chlorobenzene is obtained from a commercial vendor at a concentration of 2,000 μg/mL in methanol.

7.3.1.3 For aqueous samples, both α,α,α-trifluorotoluene and chlorobenzene are used as surrogates.

7.3.1.4 When soil samples are preserved in the field with surrogated methanol (as recommended in Alaska Method AK101), a sample of the surrogated methanol (a field blank) must be submitted to the lab with the samples so that the surrogate concentration may be verified. In these cases, the lab may opt to add a second, different surrogate at the time of analysis to assess the analytical recovery.

7.3.1.5 The Alaska method specifies using α,α,α-trifluorotoluene and/or bromofluorobenzene as a surrogate. If soil samples are submitted for the Alaska method and contain the bromofluorobenzene, then the laboratory will perform an initial calibration using that surrogate, which will be prepared at the same concentration levels as the routine surrogates.

7.3.2 Surrogate Working Standard, 30 μg/mL

7.3.2.1 A working surrogate solution is prepared for each surrogate by diluting 300 μL of the stock standard solution (Section 7.3.1) to 20.0 mL with methanol to achieve a final concentration of 30 μg/mL. For aqueous samples, the internal standard and both surrogates are combined in a single solution as described in Section 7.4.3.

7.3.2.2 Alaska Method AK101 requires the addition of a methanol solution containing the surrogate at a concentration 2.4 μg/mL to the soil samples in the field. This is referred to as “surrogated methanol.” A client submitting soil samples preserved in the field in this way must also provide a field blank containing the surrogated methanol so that the surrogate concentration may be verified at the time of analysis.

7.4 Internal Standard

7.4.1 Internal Standard (IS) Stock Solution

The internal standard used for this method is 1-chloro-4-fluorobenzene. The internal standard stock solution is obtained from a commercial source at a concentration of 2500 μg/mL in methanol.

7.4.2 IS Working Spike Solution for Soil Samples, 30 μg/mL

The internal standard working spike solution for soil samples is prepared by diluting 240 μL of the stock standard with methanol to a final volume of 20 mL to achieve a concentration of 30 μg/mL.

7.4.3 IS/Surrogate Working Spike Solution for Aqueous Samples, 30 μg/mL

The IS/surrogate working spike solution for aqueous samples is prepared by diluting 300 μL of the α,α,α-trifluorotoluene stock standard (Section 7.3.1), 300 μL of the chlorobenzene stock standard (Section 7.3.1), and 240 μL of the IS stock standard (Section 7.4.1) in a final volume of 20 mL of methanol to achieve a concentration of 30 μg/mL for each component.

NOTE: The Oklahoma Methods 8020/8015 (modified) and 8021B/GRO do not include chlorobenzene as a surrogate.

7.5 Retention Time (RT) Reference Standards

RT reference standards are used to establish retention time windows for the analytes of interest.

NOTE: The Oklahoma Methods 8020/8015 (modified) and Method 8021B/GRO do not use a separate RT reference standard. The retention time windows are updated using the first CCV of the run.

7.5.1 RT Stock Standard

The RT stock standard is purchased commercially as a pre-prepared mixture of eleven alkane and aromatic hydrocarbon compounds at the concentrations shown for the 8015 GRO method and the alkanes, C_5, C_6, C_8, C_{10}, C_{12} and C_{13} at the concentrations shown for the Method 8021B/GRO in Table 9.

7.5.2 Working RT Standard

Dilute 36 μL of the RT stock standard (Section 7.5.1) with methanol to a final volume of 1.8 mL to achieve the concentrations shown in Table 9.

7.5.3 RT Marker Standard

The internal standard, 1-chloro-4-fluorobenzene, is also used as the daily RT marker standard for making daily adjustments to the established RT windows. Since the internal standard is added to all standards, samples, and QC samples, retention time can be monitored continuously throughout the analytical sequence.

7.6 LCS and MS Spike Solutions

7.6.1 Spike Solution for Aqueous Samples, 100 µg/mL

For aqueous sample batches, the LCS is prepared by injecting 5 µL of the 100 µg/mL ICV/spike working standard solution (Section 7.2.6.2) into reagent water for a final volume of 5.0 mL. The MS and MSD are prepared similarly by injecting 5 µL of the 100 µg/mL ICV/spike working standard solution into an aliquot of the selected aqueous sample for a final volume of 5.0 mL. This results in a sample GRO concentration of 100 µg/L in the LCS, and a "spike added" GRO concentration of 100 µg/L in the MS and MSD.

7.6.2 Spike Solution for Soil Samples, 5500 µg/mL

7.6.2.1 For soil sample batches, the LCS is prepared by adding 10 µL of the 5500 µg/mL ICV/spike stock standard solution to 10 g of reagent sand before extracting into methanol. The MS and MSD are prepared similarly by adding 10 µL of the 5500 µg/mL ICV/spike stock standard solution to 10 g of the selected sample. This results in a sample GRO concentration of 5.5 mg/kg in the LCS, and a "spike added" GRO concentration of 5.5 mg/kg in the MS and MSD. When samples collected in EnCore™ samplers are analyzed, use one-half the spike volume.

7.6.2.2 The stock and intermediate level standards are stored in Teflon-sealed screw-cap/crimp cap bottles with no headspace, and kept at –10 °C to –20 °C protected from light.

7.6.2.3 The shelf life is 6 months for the stock standards, 6 months for intermediate level standards, and one month for the working level standards, which are prepared fresh monthly or more frequently as results indicate.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water Samples

8.1.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free glass 40 mL vials with Teflon-lined septa in the caps. The Teflon layer must contact the sample (zero headspace).

8.1.2 Samples are preserved with 200 µL of 50% HCl. Alaska Method AK101 specifies 200 µL of 50% HCl as preservation for volatile analytes. Oklahoma and NWTPH-Gx specify preservation with HCL to a pH<2.

8.1.3 Samples must be stored at ≤ 6°C (without freezing) and analyzed within 14 days of collection. NWTPH-Gx requires analysis within 7 days.

8.1.4 Alaska Method AK101 specifies a trip blank consisting of a contaminant-free amber glass 40 mL vial with Teflon-lined septum filled to zero headspace with purged, organic-free water preserved with the same acid as the samples.

8.2 Soil Samples

8.2.1 This procedure is designed to work with soil samples preserved and extracted in methanol. To minimize the loss of volatiles during handling in the field and in transit to the laboratory,

8.2.1.1 Method 5035, Oklahoma and NWTPH-Gx preserve high level samples in the field by adding 5.0 ± 0.5 g of soil to a pre-weighed 40 mL vial containing 10 mL of methanol.

8.2.1.2 Alaska Method AK101 requires preservation of soil samples in the field by submerging the sample in methanol to which the surrogate has been added. See Appendix A for detailed instructions for Method AK101.

8.2.2 If the methanol preservation cannot be done in the field, the lab must be supplied with an intact portion of sample in an EnCore™ sampler or in a volatile organic vial. In which case, the laboratory will perform the preservation step. The maximum holding time between collection and preservation at the laboratory is 48 hours. Per Method AK101, data from lab preserved samples must be reported as "greater than or equal to" the calculated concentration of GRO as gasoline.

8.2.2.1 The lab must also be provided with a 2-ounce glass jar containing a portion of the sample to use for screening and for moisture determination.

8.2.2.2 Samples must be stored at ≤ 6 °C and, once preserved in methanol, analyzed within 14 days of collection. Method NWTPH-Gx requires analysis within 1 week of preservation in methanol.

8.2.3 Alternatively, soil samples may be collected in 4-ounce jars packed with minimum head space and extracted with methanol at the lab and analyzed within 14 days per Method 5035 high concentration soil method.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS special instructions to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via

special instructions in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12 or more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Preparation Batch

9.3.1 A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

9.3.2 As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3.3 The Oklahoma methods require an initial MB prior to analysis and one MB and MS/MSD per 10 samples.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for water batches consists of 5 mL of reagent water, and for solids batches, 10 g of reagent sand.

Acceptance Criteria: The result for the method blank must be less than one-half the reporting limit or less than 10% of the GRO concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting or less than 10% of the lowest sample concentration.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If GRO was <u>not</u> detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample (LCS), 100 µg/L or 5.5 mg/kg

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of 5 mL of reagent water to which 5 µL of the ICV/spike working standard (Section 7.2.6.2) is added. The LCS for aqueous sample batches also functions as the ICV (second-source calibration verification). For soil sample batches, the LCS consists of 10 g of reagent sand to which 10 µL of the ICV/spike stock standard (Section 7.2.6.1) is added. The LCS is carried through the entire analytical procedure. See Section 10.8.1.2 for the addition of surrogates and internal standards.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. For aqueous sample batches where the LCS also functions as the ICV, the control limits are 85 to 115%. For soil batches, the control limits are set at ± 3 standard deviations around the historical mean and must be no wider than the limits specified in the reference methods. Alaska Method AK101 limits are summarized in Table 12. Oklahoma requires a recovery of ± 20% for waters and ± 40% for soils.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and GRO is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 100 µg/L or 5.5 mg/kg

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. (See Section 9.5 for spike amounts.) A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs will allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean. The RPD between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean.

Alaska Method AK101 limits are summarized in Table 12. Oklahoma requires a recovery of ± 20% for waters and ± 40% for soils.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, the associated LCS must be in control for the data to be reported. If there is no evidence of analytical problems and all other QC criteria are met, then qualified results may be reported and the situation must be described in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated).

9.7 Surrogate Spikes

9.7.1 The α,α,α-trifluorotoluene (TFT) surrogate standard (Section 7.3) is added to all field and QC samples prior to extraction or analysis, as appropriate. For aqueous samples, 5 µL of the IS/surrogate working spike solution (Section 7.4.3) is added to 5mL of the aqueous sample just prior to injection. For soil samples, 7.5 µL of the surrogate stock standard (Section 7.3.1) is added to 5 g of the soil sample prior to extraction with methanol.

9.7.2 For EnCore™ or Alaska Method AK101 samples, the same surrogate spike levels are used.

9.7.3 For AFCEE projects under the 3.1 and earlier QAPPs, chlorobenzene is used as the surrogate in place of TFT.

9.7.4 No surrogate is specified for the Oklahoma method so it is recommended that the analyst follow the procedure for method 8015.

NOTE: When soil samples are preserved in the field with surrogated methanol (as recommended in Alaska Method AK101), a sample of the surrogated methanol (a field blank) must be submitted to the lab with the samples so that the surrogate concentration may be verified. In these cases, the lab may opt to add a second, different surrogate at the time of analysis to assess the analytical recovery.

Acceptance Criteria: The recovery of the surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Alaska Method AK101 limits are summarized in Table 12. In addition, the RT of the surrogate must fall within the established RT window.

Corrective Action: If surrogate recoveries are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that instrument performance is acceptable. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries

may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If the surrogate recovery is outside of the established limits due to well documented matrix effects, the results must be flagged and an explanation included in the report narrative. As with matrix spike failures, some programs (e.g., USACE) may require additional analyses to confirm suspected matrix interferences. The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to re-prepare / reanalyze a sample once to demonstrate a matrix effect is reproducible.

For Alaska Method AK101 soils that are preserved in the field with surrogated methanol, if the field-added surrogate fails acceptance criteria, it may be possible to report results with appropriate narration if the laboratory-added surrogate meets acceptance criteria.

If the surrogate peak falls outside of its established RT window, it may be necessary to re-establish the center of the RT window and reanalyze affected samples. This requires re-starting the analytical sequence. (See Section 10.6.)

9.8 RT Reference Standard:

An RT reference standard containing the predominant gasoline components (see Table 9) is tested with each initial calibration to establish the retention time window. The RT reference standard is prepared by adding 5 μL of the working RT standard (Section 7.5.2) and 5 μL of the IS/surrogate working spike solution (Section 7.4.3) to 5 mL of reagent water, which is then injected into the purge-and-trap apparatus.

9.9 RT Marker Standard:

At the beginning of each day's analytical sequence, an RT reference standard is analyzed. The internal standard, 1-chloro-4-fluorobenzene, is added to the RT reference standard along with the surrogate compounds. The midpoint of the RT window for the internal standard is set for the day based on these results. See Section 10.5. If the midpoint of the RT window for the internal standard in any subsequent field sample, QC sample, or CCV slips by more than the established retention time window, then the data for that sample or standard is invalid and samples are reanalyzed as necessary.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be

completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.2 Generally, samples are screened prior to analysis by purge-and-trap GC/FID. Sample aliquots used for the definitive analysis are based on the screening data.

10.3 Gas Chromatographic Conditions

10.3.1 Recommended instrument conditions are shown in Table 10. These can be adjusted prior to calibration in order to improve performance.

10.3.2 The routine purge volume is 5 mL.

10.3.3 Instrument conditions and columns must be chosen to meet the acceptance criteria presented in this section.

10.4 Retention Time (RT) Window

10.4.1 Before establishing retention time (RT) windows, ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

10.4.2 Analyze an RT Reference Standard (Section 7.5) three times throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

10.4.3 Calculate the mean and standard deviation of the three absolute retention times for each surrogate and the internal standard.

10.4.3.1 The width of the retention time window for each analyte and surrogate peak is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period.

10.4.3.2 In those cases where the standard deviation for a particular analyte is zero, the minimum window to use is method specific:

10.4.3.2.1 8000B or 8000C: ± 0.03 minute

10.4.3.2.2 AK101: ± 0.05 minute

10.4.3.2.3 Oklahoma: ± 0.1 minute

10.4.4 For the GRO identification and quantization, the retention time window is specified by the method based on the range of hydrocarbons to be included as described below:

10.4.4.1 SW-846 Methods (8015B, 8015C and 8015D): the lower limit for the first eluting compound and the upper limit of last eluting compound.

10.4.4.2 AK101: start of C_6 through start of C_{10}

10.4.4.3 Oklahoma: start approximately 0.1 min before the retention time of MTBE and ending 0.1 min after the RT of naphthalene.

10.4.4.4 NWTPH-Gx: toluene through naphthalene; window for both sample and standards can be adjusted if interference observed in the sample.

10.4.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

10.5 Daily Retention Time Windows

10.5.1 At the beginning of each daily analytical sequence, analyze the RT marker standard (Section 7.5.3). The center of the retention time windows are adjusted based on the retention time observed for the RT marker standard and the start and endpoints for the hydrocarbon ranges.

10.5.2 A notation is made in the run sequence log to identify any standard used for the retention time verification and adjustments.

10.5.3 The Oklahoma Methods 8020/8015 (modified) and Method 8021B/GRO do not use a separate RT reference standard. The retention times are updated using the first CCV of the run.

10.6 Analytical Sequence

10.6.1 The analytical sequence starts with an RT reference standard, initial calibration (ICAL) and initial calibration verification (ICV or LCS), or with a daily continuing calibration verification (CCV). See Section 7.2.

10.6.2 For aqueous sample batches, the ICV serves as the LCS, and is followed by a method blank. For soil batches, the LCS and the method blank are analyzed following the initial calibration event or the daily CCV.

10.6.3 Samples are analyzed and are interspersed with a CCV check performed after every 12 hours or every 10 samples, whichever is more frequent.

10.6.4 If the measured concentration of any sample exceeds the highest calibration standard concentration, the sample extract must be diluted and reanalyzed. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

10.6.5 Blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination beyond established “column bleed”, the column must be baked out and subsequent blanks analyzed until the system is shown to be free from contaminants.

10.6.6 If a sample is discovered to have a high concentration, then the sample immediately following the high concentration sample must be evaluated for detection of target analytes. If target analytes are detected at or above the RL, then reanalyze the sample to rule out carryover from the high concentration sample.

10.6.7 The analytical sequence is bracketed with a closing CCV.

10.7 Aqueous Sample Analysis

10.7.1 Remove the plunger from a 5 mL syringe.

10.7.2 Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel.

10.7.3 Replace the plunger and compress the sample.

10.7.4 Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL.

10.7.5 Add 5 µL of the IS/surrogate working spike solution (Section 7.4.3) to all field and QC samples.

10.7.6 Add 5 µL of the ICV/spike working standard (Section 7.2.6.2) to the LCS, MS, and MSD.

10.7.7 This process destroys the validity of the liquid sample for future analysis. Therefore, if there is only one 40 mL vial, then transfer sample from the 40 mL vial to a 20 mL vial so that it is completely full (no headspace). This second sample is maintained only until it has determined that the first sample has been analyzed properly. If a second analysis is needed, it must be analyzed within 24 hours. Care must be taken to prevent air from getting into the 20 mL vial.

10.7.8 Attach the syringe to the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.7.9 Close both valves and purge the sample. See Table 10 for recommended purge-and-trap conditions, and refer to the instrument operation manuals for routine and non-routine maintenance.

10.8 Soil Sample Preparation

NOTE: Alaska Method AK101 requires results for soil samples to be reported on a dry-weight basis. Therefore moisture determination must accompany all soils data. The client must provide an unpreserved portion of sample for the moisture determination. The percent moisture is determined by weighing an aliquot of the sample as received and weighing it again after it has dried overnight at 105°C.

10.8.1 Preserved Soil Samples Received in Pre-Weighed Containers

NOTE: In the field, the soil sample is placed in a vial that contains surrogated methanol. The weight of the vial plus methanol is determined prior to adding the sample and is marked on the vial.

10.8.1.1 Weigh the sample container and subtract the number written on the vial from this weight to obtain the weight of the sample. Do not add any labels to the vial until this weight has been determined.

NOTE: The number marked on the vial is the weight of the vial plus the methanol.

10.8.1.2 Add 5 µL of the specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1) to each field sample, blank, LCS, MS, and MSD.

NOTE: For Alaska Method AK101 samples, the methanol used to preserve the sample in the field should already contain the appropriate surrogate, either bromofluorobenzene or α,α,α-trifluorotoluene. A second, alternate surrogate should be added in the laboratory to verify analytical accuracy and serve as check for the field-added surrogate.

10.8.1.3 Add 5 µL of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.8.1.4 Proceed to Section 10.9.1

10.8.2 Unpreserved Soil Samples Received in Pre-Weighed Containers

10.8.2.1 Weigh the sample container as received to confirm the field weights, and to determine the weight of the sample, and record the weight.

10.8.2.2 If the field weight differs by more than 0.2 gram from the laboratory-determined weight, the sample collection is considered invalid. The most common reason for disagreement in weights is extra labels added to the container after weighing in the field. However, it is possible that the weights disagree because volatile components have been lost since the time of sample collection. Contact the client before proceeding with analysis if the weights do not agree.

10.8.2.3 If the weight of the sample is ≥ 10 g, then treat the sample as "not pre-weighed" and proceed to Section 10.8.3

10.8.2.4 If the weight of the sample is less than 10 g, then add 5 mL of methanol and 3.75 µL of the specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1) to each field sample, blank, LCS, MS, and MSD.

10.8.2.5 Add 5 µL of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.8.2.6 Proceed to Section 10.9

10.8.3 Unpreserved Soils Received in Containers Not Pre-Weighed

10.8.3.1 EnCore™ Soil Samples: Obtain the tare weight of a 20 mL vial, including the cap, that contains 5 mL of methanol and 5 µL of the specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1). Extrude the sample from the EnCore™ into the vial, cap, and weigh.

10.8.3.2 Ensure that 5 µL of the specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1) is added to each field sample, blank, LCS, MS, and MSD.

10.8.3.3 Add 5 µL of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.8.3.4 If there is sufficient sample, split the sample to create a duplicate portion and extract both portions of the sample.

NOTE: To comply with Method 5035, this extraction must be done within 48 hours of collection. Some work plans may have special provisions for longer holding times for samples frozen in EnCore™ samplers, but that is not routine.

10.8.3.5 **Bulk Soils**: Bulk soil samples are submitted in packed, unweighed jars, vials, or core tubes. Transfer 10 ± 0.5 grams of soil into a prepared 20 mL vial that contains 10 mL of methanol, 7.5 µL of the specific surrogate stock standard and 10 µL of spike (if required) that has been specified in method or project requirements (Section 7.3.1) and cap the vial. This transfer must be performed very quickly to minimize loss of target analytes.

10.8.3.6 Ensure that 7.5 µL of the specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1) is added to each field sample, blank, LCS, MS, and MSD.

10.8.3.7 Add 10 µL of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.9 Aqueous and Soil Sample Analysis

10.9.1 After combining the sample, methanol, surrogate, and spikes (if appropriate), cap the vial and shake for 2 minutes.

10.9.2 Allow the sediment to settle.

10.9.3 If not analyzed immediately, these extracts must be stored at ≤ 6°C in the dark.

10.9.4 Use Table 11 to determine the volume of methanol extract to add to 5 mL of reagent water for analysis. If a screening procedure was used, use the estimated concentration to determine the appropriate methanol volume. The maximum volume of methanol that can be added to the reagent water for analysis is 100 µL. If an extract volume of less than 100 µL is used, then a volume of reagent methanol is added to total 100 µL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

10.9.5 Remove the plunger from a 5.0 mL Luerlock-type syringe equipped with a syringe valve, and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL.

10.9.6 Pull the plunger to 5.0 mL to allow volume for the addition of the sample extract and of internal standard. Add the volume of methanol extract determined from screening and a volume of methanol solvent to total 100 µL (excluding methanol in standards). Add 5 µL of the 30 µg/mL IS working spike solution (Section 7.4.2) to the water in the syringe.

10.9.7 Attach the syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.9.8 Analyze all reagent blanks on the same instrument as that used for the samples. The method blank should contain 100 µL of the methanol used to extract the organic-free reagent sand.

10.10 Calibration

Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and in the public folder, Arizona Calibration Training.

10.10.1 Initial Calibration (ICAL)

10.10.1.1 A new calibration curve must be generated after major changes to the system or when continuing calibration criteria cannot be met. Major changes include installation of new columns and changing FID jets.

10.10.1.2 The ICAL is performed using six or seven concentration levels (see Section 7.2.5 and Tables 4-6). The lowest calibration concentration is at or below the laboratory reporting limit (RL) concentration. The highest standard defines the highest sample extract concentration that may be reported without dilution.

10.10.1.3 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.10.1.4 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.10.1.4.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous;

10.10.1.4.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.10.1.4.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.10.1.4.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.10.2 The external standardization method is used for all methods except 8021B/GRO and Oklahoma which use the internal standardization method. (See Section 10.10.4.) Tabulate the area response for each calibration level

against the concentration injected. The ratio of the response to the concentration injected, defined as the calibration factor (CF), is calculated for the standard at each concentration as follows:

$$CF_i = \frac{A_{fuel}}{C_{fuel}} \qquad \textit{Equation 4}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.
A_{fuel} = Total area of the fuel calibration standard peak.
C_{fuel} = Concentration of fuel calibration standard, mg/mL

If the percent relative standard deviation (%RSD) for the average (mean) of the calculated calibration factors is less than 20%, the average calibration factor can be used for sample quantitation.

$$\text{AverageResponseFactor} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 5}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.
n = The number of calibration levels.

10.10.3 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through other options until the calibration criteria are met. Pay particular attention to the residuals noticed at the upper and lower end of the curves. This may be cause for rejection of a curve fit even if the calibration acceptance criteria are met.

10.10.3.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte or hydrocarbon range in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in Equation 4 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 6}$$

Where:

CF_i = Calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \textit{Equation 7}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 8}$$

10.10.3.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The % RSD must be ≤ 20% (AK101 upper limit is 25%).

Corrective Action: If the % RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.10.3.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). A weighted least squares regression may be used if at least three multi-point calibrations have been

performed. The weighting used is the reciprocal of the square of the standard deviation. The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \textit{Equation 9}$$

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 10}$$

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 11}$$

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.10.3.4 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum “Clarification Regarding Use of SW-846 Methods”, Attachment 2, Page 9, includes the statement “The Agency further recommends the use of a weighted regression over the use of an unweighted regression.”

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., AFCEE, DoD) require a correlation coefficient ≥ 0.995. Note that the AK102/AK103 method requires that r^2 be greater than or equal to 0.995 but method AK101 does not list a specification.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.10.3.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 12}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.10.3.6 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica-Denver.

10.10.3.6.1 Second-order regressions should be the last option. Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

10.10.3.6.2 Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH) of the expected concentration for that point.

10.10.4 Internal Standard Calibration

10.10.4.1 For 8021B/GRO and the Oklahoma Methods 8020/8015 (modified), internal standard calibration is used. Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to responses of specific standards added to the sample or sample extract prior to injection. For this method, the internal standard is 1-chloro-4-fluorobenzene, which is added to each standard, field sample, and QC sample at the time of injection.

10.10.4.2 The ratio of the peak area of the target compound in the sample extract to the peak area of the internal standard in the sample extract is compared to a similar ratio derived for each calibration standard. This ratio is the response factor (RF).

For each of the initial calibration standards, calculate the RF value for the GRO relative to the internal standard as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$ *Equation 13*

Where:

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Absolute weight of analyte purged in μg.

C_{is} = Absolute weight of the internal standard purged in μg.

10.10.4.3 The response factor is a measure of the slope of the calibration relationship and the assumption is made that the curve passes

through the origin. To evaluate the linearity of the calibration, calculate the mean response factor, the standard deviation (SD), and the relative standard deviation (RSD) as follows:

$$\text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n} \qquad \textit{Equation 14}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}} \qquad \textit{Equation 15}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100\,\% \qquad \textit{Equation 16}$$

Where:

RF_i	=	Response factor for the i^{th} calibration level.
n	=	Number of calibration levels

If the RSD of the response factors is ≤ 20%, then linearity through the origin may be assumed, and the average response factor may be used to determine sample concentrations as follows. Otherwise, a linear least-squares regression fit must be used for the calibration.

$$C_e = \frac{A_s \times C_{is}}{A_{is} \times \overline{RF}} \qquad \textit{Equation 17}$$

Where:

C_e	=	Absolute weight of the analyte or surrogate in the sample purge in μg.
A_s	=	Peak area of the analyte or surrogate.
A_{is}	=	Peak area of the internal standard.
C_{is}	=	Absolute weight of the internal standard purged in μg.
$\overline{RF}$	=	Average response factor from calibration.

10.10.4.4 To establish the least-squares regression calibration function, the instrument response (peak area) is treated as the dependent variable (y), and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b \qquad \textit{Equation 18}$$

Where:

y = Instrument response (peak area).
m = Slope of the line.
x = Concentration or amount of the calibration standard.
b = Intercept of the line.

10.10.4.5 To calculate a sample concentration or amount, the equation is solved for concentration/amount, taking the following form:

$$x = \frac{y - b}{m}$$ *Equation 19*

For the internal standard method, the regression equation is rearranged as follows:

$$\frac{A_s C_{is}}{A_{is}} = mC_s + b$$ *Equation 20*

To calcualte a sample concentration (or amount), this equation is solved for concentration (or amount) as follows:

$$C_e = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{m}$$ *Equation 21*

Where:

C_e = Concentration or amount of the target analyte in the sample purge.
A_s = Peak area for target analyte in sample purge.
A_{is} = Peak area for internal standard.
C_{is} = Concentration or amount of the internal standard.
m = Slope of the line.

10.10.4.6 The correlation coefficient, r, of the fitted line must be ≥ 0.990. Some programs (e.g., AFCEE and USACE) require r ≥ 0.995, unless approval is given in the project QAPP to use 0.990.

Method NWTPH-Gx requires a linear correlation coefficient of ≥ 0.990 with none of the standards varying from their true value by more than ± 15%.

10.10.4.7 If the ICAL %RSD or correlation coefficient linearity criteria are not met, sample analysis cannot be performed. Check that the instrument is performing properly, and adjust as needed. Check

that the standards are made correctly. After correcting any problems, prepare and reanalyze a new ICAL.

10.10.5 Second-Source Initial Calibration Verification (ICV)

Analyze a 100 μg/mL second-source verification standard after each ICAL. For aqueous sample batches, the ICV is also the LCS (Section 7.2.6.2). For soil sample batches, the ICV is separate from the soil LCS.

Acceptance Criteria: The percent difference (%D) between the measured concentration for this standard and the expected concentration must be within ± 15%. When the ICV also functions as the LCS for aqueous soil batches, the percent recovery for the LCS must be within 85 to 115%. Methods 8015C and 8015D allow up to ± 20%D.

Corrective Action: If the %D or % recovery for the second-source verification fails acceptance criteria, then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result also fails acceptance criteria, then re-prepare the verification standard, and/or re-prepare and rerun the ICAL.

The %D and % recovery are calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Expected Conc}}{\text{Expected Conc}} \times 100\% \qquad \textit{Equation 22}$$

$$\%\,\text{Recovery} = \frac{\text{Conc Recovered}}{\text{Conc Added}} \times 100\% \qquad \textit{Equation 23}$$

10.10.6 Continuing Calibration Verification (CCV)

Analyze a mid-point calibration standard every 12 hours or after 20 sample analyses, whichever is more frequent, and upon completion of sample analyses to bracket the run. Method 8000C requires two levels for the CCV when a quadradic fit is used for the ICAL. The concentrations of the CCVs in this case are near the midpoint of the calibration curve and near the RL. (See Section 7.2.2.5) It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

Acceptance Criteria: The result for each analyte must be within ± 15% of the expected value. Methods 8015C, 8015D, Oklahoma, and NWTPH-Gx allow a ± 20% drift. AK101 allows ± 25% drift.

Corrective Action: If one or more analytes fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to original settings or perform routine maintenance. This may include clipping the column, changing the liner, or other minor instrument adjustments. Inject another aliquot of the CCV standard.

If the response for any analyte is still not within ± 15%, then a new initial calibration must be performed. Any samples injected after the last valid CCV standard must be re-injected.

11.0 Calculations / Data Reduction

The concentration of GRO in the sample is determined by calculating the absolute weight of analyte purged, from a summation of peak response for all chromatographic peaks, resolved and unresolved, eluting between 2-methylpentane (C_6) and 1,2,4-trimethylbenzene (C_9), using the calibration curve or the response factor determined in Section 10.10 For Alaska Method AK101, the range is defined as all chromatographic peaks, resolved and unresolved, eluting between the peak start time of hexane (C_6) and the peak start time of decane (C_{10}). Second column confirmation is not normally performed for this method as confirmation of chemical identity is not part of the analysis.

11.1 Calculation of Sample GRO Concentration

GRO in the sample extract (C_e) is calculated using the calibration function as described in Section 10.10 The GRO concentration in the original sample is calculated as follows:

11.1.1 GRO Concentration in Aqueous Samples

$$C_s = \frac{C_e}{V_s} \times DF \qquad \textit{Equation 24}$$

Where:

C_s = Concentration of GRO in the original sample in μg/L.
C_e = Absolute weight of GRO in sample purged in μg.
V_s = Volume of sample purged in liters.
DF = Dilution factor.

11.1.2 GRO Concentration in Soil Samples

$$C_s = C_e \times \frac{V_m \times DF}{W_s \times V_p} \qquad \textit{Equation 25}$$

Where:

C_s = Concentration of GRO in the original sample.
C_e = Absolute weight of GRO in sample extract purged in μg.
V_m = Volume of methanol extract in μL (e.g., 10 mL = 10,000 μL).
W_s = Weight of sample extracted in kg. For Alaska Method AK101 and as specified by project requirements, the dry weight is used.
V_p = Volume of extract actually purged in μL.
DF = Dilution factor.

NOTE: For Alaska Method AK101, and as specified by project requirements, the percent moisture of a soil sample is calculated by subtracting the weight after drying from the original sample weight, dividing that by the original sample weight, and expressing the result as a percent.

NOTE: For work performed for the state of Alaska, the volume of methanol used for extraction must be adjusted for the moisture content of the sample by the following equation:

$$V_t = V_m + M * W_s/100 \qquad \textit{Equation 26}$$

Where:

Vt = final extract volume, corrected for moisture (mL)
Vm = volume methanol used for extraction (mL)
M = moisture content of the sample (%)
Ws = aliquot of sample extracted (g)

11.2 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing.

11.2.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.2.2 All manual integrations must be justified and documented. See DV-QA-011P requirements for manual integration

11.2.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.2.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the before and after chromatograms to the same scale and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.3 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.3.1 Perform a Level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.3.2 Submit the data package and review checklist to the peer reviewer for the Level 2 review. All manual integrations must be evaluated by the peer reviewer and initialed on the manual integration summary report. The Level 2 review is documented on the review checklist initiated at the Level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

12.2 Initial Demonstration of Capability

IDOCs must be performed initially, before samples are analyzed, and must be repeated on an annual basis. Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

12.2.1 Initial demonstration of ability for Oklahoma method

12.2.1.1 Analyze 7 replicates of organic free water spiked with the gasoline component standard at a concentration of 2 ug/L for each individual component with recoveries for all components within ± 30% of the known concentration and precision of all replicates within ± 20%.

12.2.1.2 Analyze 7 replicates of GRO-free sand spiked with the gasoline component standard at the reporting limit (50 ug/kg) for each individual component with recoveries for all components within ± 40% of the known concentration and precision of all replicates within ± 20%.

12.3 Training Qualification

The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and DV-HS-001P, “Waste Management Program.”

The following waste streams are produced when this method is carried out:

- Methanol extract vial waste – Expired Extract Vials, Waste Stream A
- Purge-and-trap aqueous waste – Flammable Solvent Waste, Waste Stream C
- Solvent rinse waste – Flammable Solvent Waste, Waste Stream C
- Expired Chemicals/Reagents/Standards – Contact the Waste Coordinator

  Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

15.1.3 Method 8015B, Nonhalogenated Organics Using GC/FID, Revision 2, December 1996.

15.1.4 Method 8015C, Nonhalogenated organics by Gas Chromatography, Revision 3, February 2007.

15.1.5 Method 8015D, Nonhalogenated organics by Gas Chromatography, Revision 4, June 2003.

15.1.6 Method 5000, Sample Preparation for Volatile Organic Compounds, Revision 0, December 1996.

15.1.7 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December 1996

15.1.8 Method 5030C, Purge-and-Trap for Aqueous Samples, Revision 3, May 2003.

15.1.9 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December 1996.

15.1.10 Method 5035A, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July 2002.

15.2 American Petroleum Institute "Method for the Determination of Gasoline Range Organics," Draft Revision 4- August 18, 1992, prepared by Rocky Mountain Analytical for the American Petroleum Institute.

15.3 Alaska Method AK101, "For the Determination of Gasoline Range Organics", Version 04/08/02.

15.4 Oklahoma Methods 8020/8015 (modified) Gasoline Range Organics (GRO), Revision 4.0, 02/24/96.

15.5 NWTPH-Gx, "Volatile Petroleum Products Method for Soil and Water", Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

16.0 Method Modifications:

Item	Method	Modification
1	AK101	The Alaska method recommends bromoflurobenzene and α,α,α-trifluorotoluene as surrogates. This method allows the use of alternate surrogates as long as they are non-polar, purgeable from water, elute prior to the start of C_{11}, and do not co-elute with any significant component of gasoline. This SOP uses chlorobenzene in place of bromofluorobenzene.
2	AK101	Method AK101 suggests a retention time standard consisting of n-hexane and n-decane. This SOP uses a standard mixture that includes both alkanes and aromatic hydrocarbons over the carbon range of the method.
3	NWTPH-Gx	NWTPH-Gx suggests the use of 1,4-difluorobenzene or bromofluorobenzene but also allows for the use of other surrogates as long as these compounds are non-polar, purgeable from water and do not coelute with any significant component of gasoline. α, α, α-Trifluorotoluene is used in place of the method suggested surrogates.
4	OK Methods	Standard soil RL of 1.2 mg/kg for GRO and 0.050 mg/kg for individual components is higher than the method specified RL of 0.1 mg/kg for GRO and 0.01 mg/kg for individual components. The laboratory MDLs do not support the method RL. The Oklahoma method identifies a direct purge of soil samples unless dilution is required and then a methanol extraction is performed. The lab uses the methanol extraction for all samples.
5	OK Methods	The spike level used by the laboratory to fulfill Oklahoma's requirement for the analysis of 7 replicates for demonstration of ability to generate acceptable accuracy and precision for soil has been increased to 50 ug/kg. The method requirement is to spike at the method reporting limit of 10 ug/kg for individual components. The laboratory MDLs do not support the method RL and the spike level used is the laboratory RL for the individual components.

17.0 Attachments

Table 1: Typical Commercial GRO/BTEX Calibration Standard

18.0 Revision History

- Revision 7.1, dated 18 July 2011
    - Revised Oklahoma IDOC procedure (section 12.2.1) to increase spike level for Soil IDOC to 50 ug/kg, the laboratory RL for the individual components.
    - Clarified method modification 4 and added method modification 5.
- Revision 7, dated 11 April 2011
    - Combined with SOP DV-GC-0028, Gasoline Range Organics (GRO) by GC/FID SW846 Method 8015C to provide a comprehensive SOP that covers GRO by Methods 8015B, C and D, 8021B and the state methods of Alaska, Oklahoma and Washington.
    - Removed references to CA LUFT Manual and method
    - Removed LCSD requirement except as needed based on TA-Denver policy and client request.
    - Included both internal and external standard calibration discussions
    - Updated formatting
    - Removed grand mean of %RSD for continuing calibration acceptance criteria
    - Included Oklahoma IDOC protocol
    - Expanded references section to include all methods in SOP including prep methods.
    - Updated tables to include each set initial calibration standards based on individual method requirements.
    - Added example chromatograms for RT Marker Standard and GRO standard

- Revision 6.1, dated 12 March 2010
    - Updated implementation date
    - Added section 6.3
- Revision 6, dated 19 June 2009
    - Added references and method modifications for Washington's NWTPH-Gx
    - Updated formatting
    - Added CCV criteria (section 10.9.4)
- Changes from Previous Versions
    - Revision 5, March 2008 – changed to TestAmerica format and name.
    - Incorporated Alaska Method AK101 throughout this SOP.
    - Reformatted SOP to include change in name to TestAmerica.
    - Modified Table 4 to include the AK101 RT Markers.

TABLE 1.

TYPICAL COMMERCIAL GRO/BTEX CALIBRATION STANDARD

NOTE: The following formulation is an example of a commercially available GRO/BTEX standard. In this case, the formulation is that of AccuStandard GA-001-20X-BTEX. The components are in a methanol solution.		
Component	**Stock Standard Concentration (μg/mL)**	**Intermediate Standard Concentration (μg/mL)**
Benzene	78	1.56
Ethylbenzene	320.5	6.41
Gasoline	10,025.0	200.5
m- & p-Xylenes	381.2	7.62
o-Xylene	137.7	2.75
Toluene	97.0	1.94

TABLE 2.

TYPICAL COMMERCIAL ALASKA METHOD AK101 GRO CALIBRATION STANDARD

NOTE: The following formulation is an example of a commercially available GRO standard for the Alaska method AK101. In this case, the formulation is that of AccuStandard AK-101GSC-R1. The components are in a methanol solution.		
Component	**Stock Standard Concentration (μg/mL)**	**Intermediate Standard Concentration (μg/mL)**
Gasoline	744055.2	200

TABLE 3.

TYPICAL COMMERCIAL GRO/BTEX CALIBRATION STANDARD FOR OKLAHOMA METHODS 8020/8015 (MODIFIED)

NOTE: The following formulation is an example of a commercially available GRO/BTEX standard for the Oklahoma Methods 8020/8015 (modified). In this case, the formulation is that of Ultra UST-100. The components are in a methanol solution.

Component	Stock Standard Concentration (µg/mL)	Intermediate Standard Concentration (µg/mL)
1,2,4-Trimethylbenzene	1000	20
1,3,5-Trimethylbenzene	1000	20
Benzene	1000	20
Ethylbenzene	1000	20
Gasoline	10,000	200
m- & p-Xylenes	1000	20
Methyl t-Butyl Ether (MTBE)	1000	20
Naphthalene	1000	20
o-Xylene	1000	20
Toluene	1000	20

TABLE 4.

METHOD 8015 GRO/AK101

Calibration Levels (µg/L)						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
GRO C_6-C_{10} C_5-C_{12} C_6-C_{12}	20	50	100	200	500	1000
Chlorobenzene α,α,α-Trifluorotoluene 1-Chloro-4-fluorobenzene	3	7.5	15	30	75	150

Table 5

METHOD 8021-GRO

CALIBRATION LEVELS
(µg/L)

	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Benzene	0.25	0.5	2.5	5	10	20	30
Ethylbenzene	0.25	0.5	2.5	5	10	20	30
m- & p-Xylene	1.0	2.0	10.0	20	40	80	120
o-Xylene	0.5	1.0	5.0	10	20	40	60
Toluene	0.75	1.5	7.5	15	30	60	90
MTBE	1.0	2.0	10.0	20	40	80	120
1,2,4-Trimethylbenzene	0.5	1.0	5.0	10	20	40	60
Gasoline (C_6-C_{10})	6	12.0	60.0	120	240	480	720
α,α,α-Trifluorotoluene (surrogate)	0.75	1.5	7.5	15	30	60	90

TABLE 6.

OKLAHOMA GRO METHOD

CALIBRATION LEVELS

Calibration Levels (µg/L)						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
1,2,4-Trimethylbenzene	0.5	5	10	20	50	100
1,3,5-Trimethylbenzene	0.5	5	10	20	50	100
Benzene	0.5	5	10	20	50	100
Ethylbenzene	0.5	5	10	20	50	100
m- & p-Xylene	0.5	5	10	20	50	100
Methyl t-Butyl Ether (MTBE)	0.5	5	10	20	50	100
Naphthalene	0.5	5	10	20	50	100
o-Xylene	0.5	5	10	20	50	100
Toluene	0.5	5	10	20	50	100
Gasoline	5.0	50	100	200	500	1000
Surrogate	0.75	7.5	15	30	75	150

TABLE 7.

TYPICAL COMMERCIAL 8021B/GRO CALIBRATION STANDARD

NOTE: The following formulation is an example of a commercially available 8021B/GRO standard). In this case, the formulation is that of Ultra CUS-1699A. The components are in a methanol solution.

Component	Stock Standard Concentration (µg/mL)	Intermediate Standard Concentration (µg/mL)
1,2,4-Trimethylbenzene	1000	20
2,2,4-Trimethylpentane	1500	30
2-Methylpentane	1500	30
Benzene	500	10
Ethylbenzene	500	10
Gasoline	12,000	240
m- & p-Xylenes	2000	40
Methyl t-Butyl Ether (MTBE)	2000	40
n-Heptane	500	10
o-Xylene	1000	20
Toluene	1500	30

Table 8.

TYPICAL COMMERCIAL GRO/BTEX SECOND-SOURCE STANDARD

NOTE: The following formulation is an example of a commercially available GRO/BTEX second-source standard. In this case, the formulation is that of Restek 30237. The components are in a methanol solution.

Component	Stock Standard Concentration (µg/mL)	8015 GRO Working Standard Concentration (µg/mL)	8021B/GRO Working Standard Concentration (µg/mL)
Benzene	80	1.47	8
Ethylbenzene	94	1.72	9.4
Isopropylbenzene	12	0.22	1.2
m- & p-Xylenes	327	6.00	32.7
Methyl t-Butyl Ether (MTBE)	124	2.27	12.4
Naphthalene	25	0.46	2.5
o-Xylene	130	2.38	13
Toluene	399	7.32	39.9
Unleaded gasoline composite (compliant with Alaska Method AK101)	5500	100.8	550

TABLE 9.

TYPICAL RT STOCK STANDARDS

NOTE: The following formulations are examples of commercially available stock standards (in methanol)

Table 9A

TYPICAL RT REFERENCE STANDARD – Method 8015		
Component	**Stock Standard Concentration (µg/mL)**	**Working Standard Concentration (µg/mL)**
1,2,4-Trimethylbenzene (C_9)	1000	20
2,2,4-Trimethylpentane (C_8)	1500	30
2-Methylpentane (C_6)	1500	30
Benzene (C_6)	500	10
Ethylbenzene (C_8)	500	10
m-Xylene (C_8)	1000	20
Methyl t-Butyl Ether (MTBE)	2000	40
n-Heptane (C_7)	500	10
o-Xylene	1000	20
p-Xylene	1000	20
Toluene (C_7)	1500	30

Table 9B

Typical RT Marker Standard – AK101		
Component	**Stock Standard Concentration (ug/mL)**	**Working Standard Concentration (µg/mL)**
n-Pentane (C_5)	2000	50
n-Hexane (C_6)	2000	50
n-Octane (C_8)	2000	50
n-Decane (C_{10})	2000	50
n-Dodecane (C_{12})	2000	50
n-Tridecane (C_{13})	2000	50

TABLE 10:

RECOMMENDED INSTRUMENT CONDITIONS

Purge-and-Trap Apparatus

Purge gas	Nitrogen or Helium
Purge gas flow rate	25-40 mL/min.
Purge temperature	30 °C
Desorb temperature	180 °C
Backflush inert gas flow	180 mL/min.
Desorb time	2 minutes
Purge time	11 minutes

GC Conditions

Helium Column Pressure	20 psi
Initial Column Temperature	40 ºC for 2 minutes
First Temperature Ramp	5 ºC/minute & hold at 110 ºC for 8 minutes
Second Temperature Ramp	8 ºC/minute to 190 ºC
Third Temperature Ramp	10 ºC/minute to 210 ºC & hold for 1 minute
FID Temperature	320 ºC

TABLE 11:

AMOUNT OF METHANOL EXTRACT NEEDED FOR ANALYSIS OF SOILS

GRO Approximate Concentration (µg/g) [a]	Volume of Methanol Extract (µL) [b]
0 – 500	100
500 – 2000	50
2000 – 5000	10
≥ 5000	100 µL of 1/50 dilution [c]

Calculate the appropriate dilution factor for concentrations exceeding the concentrations in this table.

[a] This value may be based on a screening result or historical knowledge.

[b] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 µL for each blank, sample, and control.

[c] Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

TABLE 12.

Alaska Method AK101 Quality Control Acceptance Criteria

Analyte	Spike Concentration		Control Limits	
	Water (mg/L)	Soil (mg/kg)	% Recovery	RPD (%)
GRO in Lab-Fortified Blanks[1]	0.1 – 1.0	5 - 100	60 - 120	20
Laboratory Sample[2] Surrogate Recovery (α,α,α-trifluorotoluene or bromofluorobenzene)	0.05	2.5	60 - 120	---
Field Sample Surrogate Recovery (α,α,α-trifluorotoluene or bromofluorobenzene)	0.05	2.5	50 - 150	---
Continuing Calibration	1.0	---	75 - 125	---
Calibration Verification	1.0	---	75 - 125	---

[1] This is the same as the TAL Denver laboratory control sample (LCS).

[2] Laboratory samples are control samples that are spiked in the laboratory, i.e., LCS, method blank, MS, and MSD.

The quality control criteria listed in this table represent the minimum acceptable levels, using highly organic soil matrices. Higher performance may be required on some projects.

APPENDIX A –

Sample Collection Instructions for Alaska Method AK101 GRO/BTEX

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TestAmerica Denver
Volatile (AK101 GRO/BTEX) Sample Collection Instructions

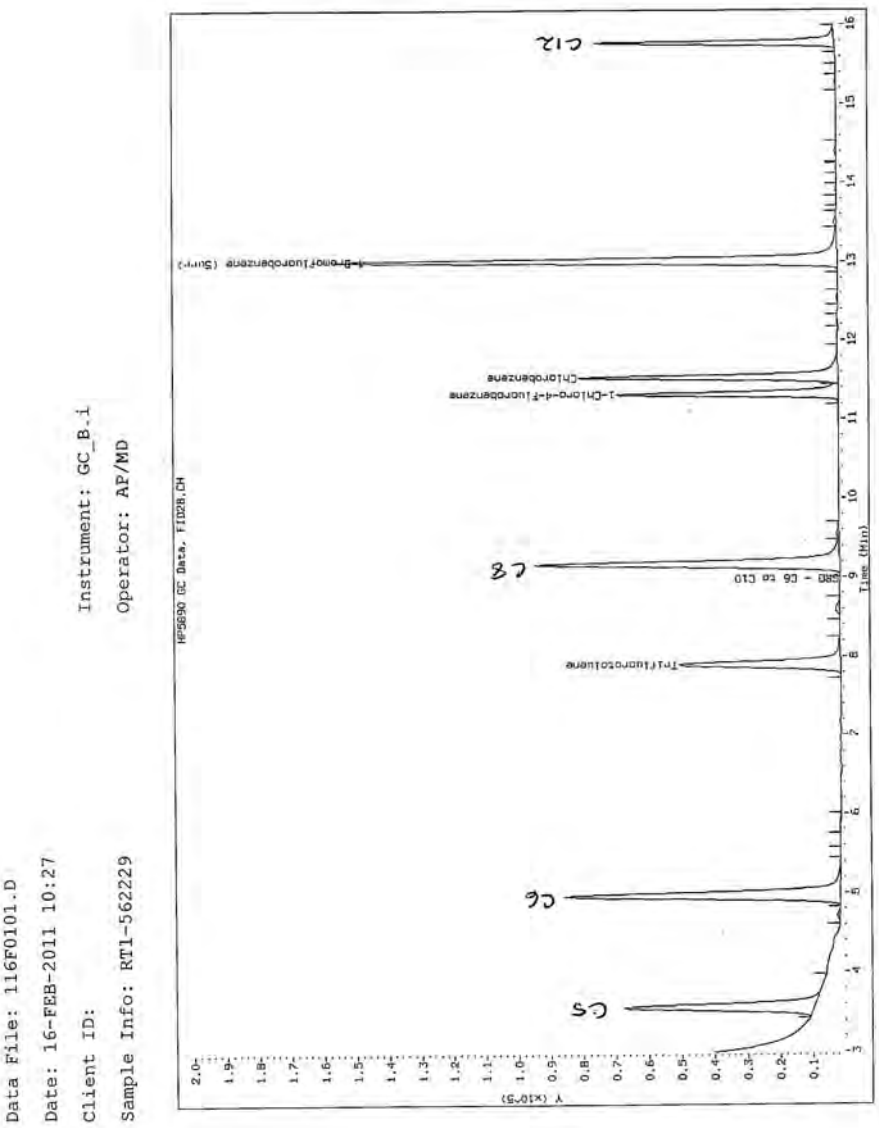
Soil Samples:

1. Soil samples should be collected in 4-oz amber jars that are pre-tared. Using a pre-tared jar ensures that the lab can very accurately determine the sample mass. TestAmerica Denver proves these containers.
2. The sample should be collected with minimum disturbance.
3. **The recommended amount of sample to collect is 25 to 50g, which is approximately 1 inch of soil inside the jar**.
4. The vial containing 25-mL of methanol/surrogate solution (also provided by TestAmerica Denver) is then carefully poured into the jar.
5. **The soil sample MUST be completely submersed in the methanol/surrogate solution**. If too much sample is collected and therefore the sample is not completely submersed in the methanol/surrogate solution, sample integrity has been compromised and the lab will issue the final report with a qualifier for the sample.
6. Samples must be refrigerated or iced to 0-6°C.
7. A trip blank (provided by the lab) must accompany each shipping container and should be stored with the field samples.
8. An additional sample of the same soil to be analyzed for GRO should be collected into a 4-oz unpreserved amber jar for percent moisture analysis in order to report results on a dry weight basis.

Aqueous Samples:

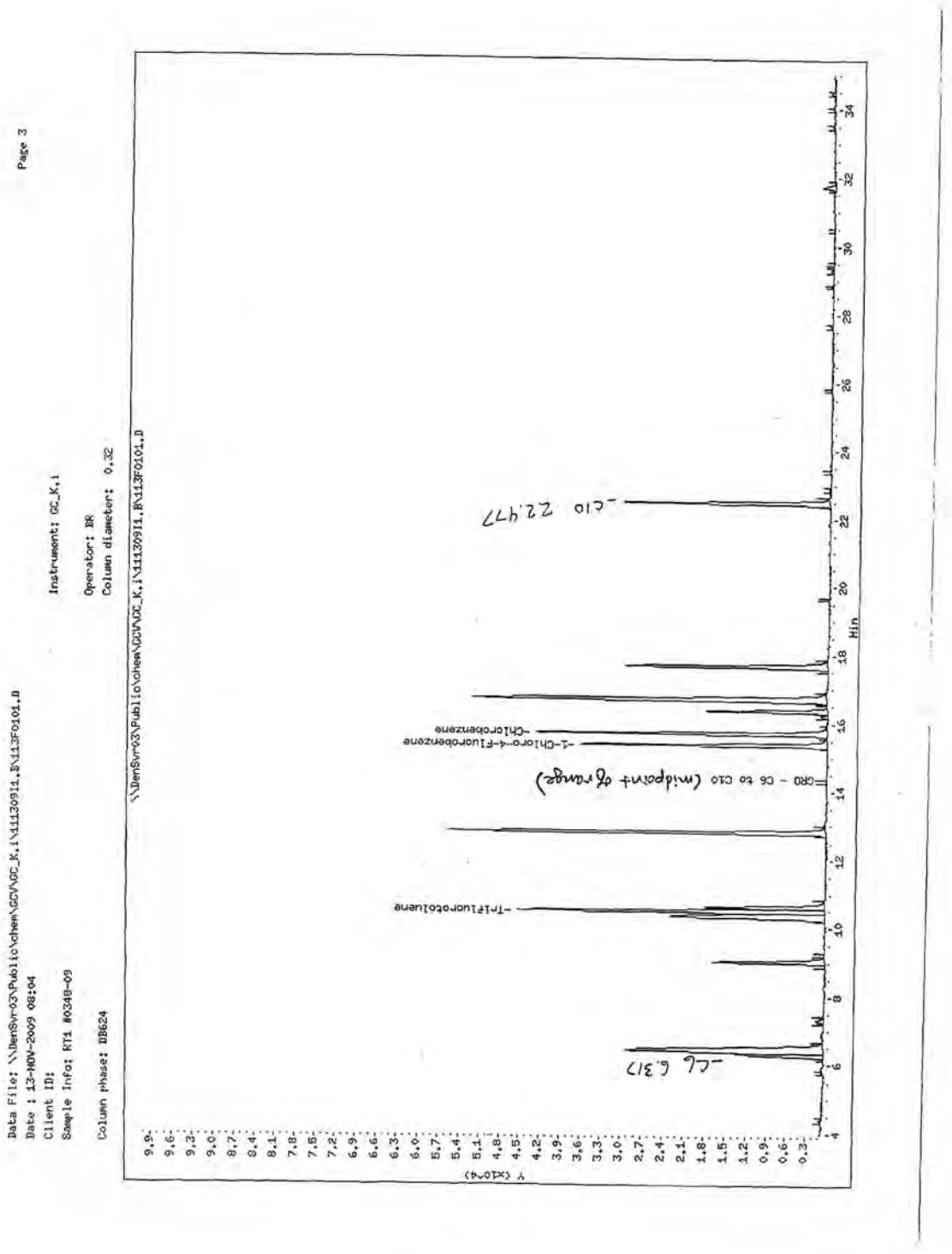
1. Aqueous samples should be collected in 40-mL HCl preserved glass VOA vials. TestAmerica Denver provides these containers. Three vials per sample are required.
2. Samples should be collected without agitation and **WITHOUT HEADSPACE**.
3. Samples must be refrigerated or iced to 0-6°C.
4. An aqueous trip blank (provided by the lab) must accompany each shipping container and should be stored with the field samples.

Figure 1
Sample Chromatogram for RT Marker Standard



Note: Actual retention times will vary by instrument and column.

Figure 2
Sample Chromatogram for GRO Standard



Note: Actual retention times will vary by instrument and column.

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Title: Polychlorinated Biphenyls (PCBs) by GC/ECD [SW846 Methods 8082 and 8082A]

Approvals (Signature/Date):

Dennis Jonsrud 5-22-12 Date Technical Manager	Adam Alban 31 May 12 Date Health & Safety Manager / Coordinator
John P. Morris 5/21/12 Date Quality Assurance Manager	Robert C. Hanisch 5/31/12 Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors using the methodology prescribed in EPA SW-846 Method 8082 and 8082A.

1.2 This procedure is applicable to the analysis of extracts of aqueous, solid, and oil samples. When utilized for the analysis of oils, additional cleanup procedures may be required. This procedure also defines the conditions required when using a large volume injection.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOPs DV-OP-0006, DV-OP-0007, DV-OP-0015, DV-OP-0016 and DV-OP-0023 for sample preparation procedures. Refer to SOP DV-OP-0012 for waste dilutions.

1.4 Additional information is provided in this SOP for the inclusion of the analysis of polychlorinated terphenyls (PCT) by the same protocols used for the determination of Aroclors.

1.5 This SOP does not include the determination of the concentration of PCB congeners.

1.6 Analytes, Matrix(s), and Reporting Limits

1.6.1 Tables 1 and LVI-1 list the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

2.0 Summary of Method

2.1 Preparation

2.1.1 Aqueous Samples

PCBs are extracted from a one-liter aqueous sample with methylene chloride using a separatory funnel (SW-846 Method 3510). The extract is evaporated to approximately 25 mL and exchanged to hexane. The final extract volume is 10 mL, however depending on special client requirements the final extract volume can also be 1 mL or 5 mL. The extraction procedure is detailed in SOP DV-OP-0006.

2.1.2 LVI Aqueous Samples

PCBs are extracted from a 35 mL aqueous sample with 2 mL of hexane (SW-846 Method 3511). The extraction procedure is detailed in SOP DV-OP-0023.

2.1.3 Solid Samples

PCBs are extracted from solid materials using either sonication or microwave extraction. If sonication extraction is selected the samples are extracted with a 50:50 Acetone:Methlyene Chloride mixture, concentrated down to approximately 25 mL, exchanged with hexane, and brought to a 10 mL final volume. See DV-OP-0016 and DV-OP-0007 for details. If microwave extraction is selected the samples are extracted with a 50:50 Acetone:Hexane mixture, and concentrated down to a 10 mL final volume. See DV-OP-0015 and DV-OP-0007 for details.

2.1.4 Oil Samples

Oil samples are typically prepared by diluting 1 gram of sample to a final volume of 10 mL with hexane. The extraction procedure is detailed in SOP DV-OP-0012.

2.1.5 Wipe Samples

Wipes are typically collected using either filter paper or gauze. These samples can then be extracted using the procedure outlined in SOP DV-OP-0016.

2.1.6 Cleanup Procedures

Cleanup options are discussed in Section 4 below. Instructions for performing various cleanup procedures are detailed in SOP DV-OP-0007.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an external standard calibration. Second column confirmation is only performed when requested by the client or as a program requirement. The presence of multiple peaks in the sample serves as confirmation of analyte presence.

3.0 Definitions

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has

12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 42% by weight chlorine.

NOTE: Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content, but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

3.3 AR1660: Laboratory designation for the mixture of Aroclors 1016 and 1260.

3.4 AR2154: Laboratory designation for the mixture of Aroclors 1221 and 1254.

3.5 AR3262: Laboratory designation for the mixture of Aroclors 1232 and 1262.

3.6 AR4268: Laboratory designation for the mixture of Aroclors 1242 and 1268.

3.7 Polychlorinated Terphenyls: Polychlorinated terphenyls (PCTs) are chemically related to PCBs with the exception that PCTs have an additional phenyl group. The PCTs included in this analysis are AR 5432, AR 5442, and AR 5460. The preparation and analysis is treated the same as for the PCB Aroclor analysis.

4.0 Interferences

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP DV-OP-0007.

All QC is brought through the cleanup process and reported with the samples. An aliquot of all samples and QC is set aside and not brought through the cleanup process. If the QC is out of criteria then the QC that wasn't brought through the cleanup process will be analyzed and used to verify the batch for the samples not brought through clean-up.

4.2 Sulfur will interfere and can be removed using procedures described in SOP DV-OP-0007.

4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Any affected samples are re-analyzed.

4.4 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector.

4.4.1 Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.4.2 Single-component chlorinated pesticides, if present, may co-elute with individual PCB congeners and interfere with the identification and/or quantitation of the aroclors. This can be addressed by analyzing a chlorinated pesticide mixed standard prior to an initial calibration to identify where potential interferences might occur.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (M-E-001 DV), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of

the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain of the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height.

6.2 Computer Software and Hardware

6.2.1 Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Columns

6.3.1 Primary Column: CLPI, 30 m x 0.32 mm id, 0.5 µm coating.

6.3.2 Secondary Column: CLPII, 30 m x 0.32 mm id, 0.25 µm coating.

6.3.3 Additional columns that can be used for confirmation include 30m x 0.32mm id HP-5 or HP-1701.

6.4 Supplies

6.4.1 Autosampler vials, crimp caps with PTFE-faced septa.

6.4.2 Y-splitter, septa, guard columns, ferrules, Siltek injection port liners, Siltek glass wool.

6.4.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4.4 Various class A volumetric flasks from 5 mL to 250 mL.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.2 Hexane, pesticide grade. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.2 Stock Standards

7.2.1 All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.2.2 All standards must be refrigerated at 0-6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.3 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.2.4 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner if comparison with check standards indicates a problem.

7.3 PCB and Surrogate Stock Calibration Standards

7.3.1 Stock A

For each of the Aroclors listed in Tables 1 and LVI-1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in pesticide-grade hexane (or in some cases, isooctane) at a concentration of 1,000 µg/mL. The current primary stock source is Accu Standard (AR1221/C-221S-H-10x; AR1016/C-216S-H-10X; AR1232/C-232S-H-10X; AR1242/C-242S-H-10X; AR1248/C-248S-H-10X; AR1254/C-254S-H-10X; AR1260/C-260S-H-10X; AR1262/C-262S-H-10X; AR1268/C-268-H-10X).

7.3.2 Surrogate Stock B

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, each at a concentration of 200 µg/mL. The current surrogate source is Accu Standard CLP-032-R-Accu.

7.3.3 PCT Stock

A commercially prepared stock standard solution is obtained that contains the individual PCT compounds at a concentration of 35 ug/mL in hexane. The current vendor is Accustandard and the catalog numbers are AR 5432 T432S, AR 5442 T442S, and AR 5460 T460S.

7.4 Intermediate and Working Level Calibration Standard Solutions

7.4.1 Stock C (Level 6 Calibration) Standard Solutions

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 6) calibration standard.

Stock C	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.1 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	1.0
	0.1 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	1.0
	0.025 mL of surrogate Stock B	200		TCMX	0.05
				DCB	0.05

Stock C	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_2154	0.1 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	1.0
	0.1 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	1.0
AR_3262	0.1 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	1.0
	0.1 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	1.0
AR_4268	0.1 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	1.0
	0.1 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	1.0
AR_1248	0.1 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	1.0

7.4.2 AR_1660 Calibration Levels

A total of 7 calibration standards are prepared for AR_1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the AR_1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.25	10	0.025	0.00125
2	0.5	10	0.050	0.0025
3	1.0	10	0.10	0.005
4	2.5	10	0.25	0.0125
5 (CCV)	50.0	100	0.50	0.025
6	7.5	10	0.75	0.0375
7 (Stock C)	--	--	1.0	0.0500
* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				

7.4.3 Working Single-Point PCB Calibration Standards

The Level 5 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.4.4 Polychlorinated Terphenyl Calibration Levels

A total of 7 calibration standards are prepared for PCTs as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the PCTs. The level 7 standard is prepared from the stocks described in section 7.3.3 by diluting 1 mL of the stock to 35 mL final volume with hexane. The final concentration of the level 7 standard is 1.0 ug/mL. In all cases, measured volumes of the Level 7 standard are

diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Level 7 Used (mL)	Final Volume (mL)	Final PCT Conc (µg/mL)
1	0.25	10	0.025
2	0.5	10	0.05
3	1	10	0.10
4	2.5	10	0.25
5 (CCV)	5	10	0.50
6	7.5	10	0.75

7.5 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.5.1 Second-Source Stock A' Aroclor Standard Solutions

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 100 µg/mL. A second source may be obtained for the other Aroclors, if necessary. The current second source is Ultra Scientific (AR1221/PP-291; AR1016/PP281; AR1232/PP301; AR1242/PP311; AR1248/PP341; AR1254/PP-351; AR1260/PP362; AR1262/PP370; AR1268/PP380.

7.5.2 Second-Source Surrogate Stock B' Standard Solution

A commercially prepared solution is obtained containing TCMX and DCB each at a concentration of 200 µg/mL. The current second source surrogate is Ultra Scientific ISM-320.

The working level second-source ICV standard is prepared by combining 0.025 mL of Aroclor 1016 Stock A', 0.025 mL of Aroclor 1260 stock A', and 0.00625 mL of surrogate Stock B', and diluting to a final volume of 10 mL with pesticide-grade hexane. This results in a concentration of 0.25 µg/mL for each of the Aroclors and 0.125 µg/mL for each surrogate. If a second source verification standard is prepared for any of the Aroclors other than the AR_1660 mixture, the surrogates are not added.

7.5.3 PCT Second Source Stock and working level.

A commercially prepared solution of each of the PCT mixes is obtained from a different vendor and typically prepared at a concentration of 100 ug/mL in hexane. The current second source vendors and catalog numbers are AR 5432 Chem Services F290RPS, AR 5442 Chem Services F860RPS, and AR 5460 Chem Services F292RPS.

The working level PCT standard is prepared at a concentration of 0.25 ug/mL by diluting 0.025 mL of each stock to a final volume of 10 mL.

7.6 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 5 initial calibration standard, as shown in the table in Section 7.4.2.

7.7 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL Standard.

7.8 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

NOTE: The LCS/MS spiking solution is prepared and used as part of the scope of the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015, and DV-OP-0016. The following information is provided for reference only.

The working level LCS solution is made from a source different from the source used to make the primary calibration standards. In general it is made up at a concentration of 2 µg/mL in a water-soluble solvent such as acetone. For oil samples extracted by waste-dilution, the standard is made in hexane. The standard contains Aroclors 1016 and 1260 only. Typically 1 mL of this standard is added to 1 liter of water samples, 30 g of soil samples, or 1 g of oil samples. The current LCS vendor is Ultra Scientific PPM-8082 at a concentration of 1000ug/ml. The solution is prepared by diluting 0.5 ml of this stock into 250 ml with acetone solvent.

7.9 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. Matrix spike samples are prepared by adding 1.0 mL of the working solution to a second one-liter aliquot of the selected aqueous sample, or to a 30-gram subsample of the selected soil sample. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

7.10 Surrogate Spike Solution

7.10.1 Stock Surrogate Spike Solution:

A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased.

7.10.2 Working Surrogate Spike Solution

NOTE: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015 and DV-OP-0016. The following information is provided for reference only.

The working level surrogate solution is made up to contain DCB and TCMX at a concentration of 0.2 µg/mL. For water and soil samples the solution is made in a water-soluble solvent like acetone. For all oil samples extracted by waste dilution the solution is made in hexane.

7.11 Primer Mix

The primer mix typically consists of a mixture of CCV standards and/or old calibration standards. The concentrations of the components of the primer mix are not critical. The primer mix is injected one or more times prior to analyzing standards and samples to ensure that the chromatographic system is stable, i.e., that retention times are reproducible.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Water[1]	Amber glass	1 Liter	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846
Water[3]	3x40 mL vial	40 mLs	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846
Solid	Glass	8 oz	Cool, $\leq 6^{o}C$	1 Year to extraction 40 days to analysis	SW-846

[1]To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

[2]California, Connecticut, Pennsylvania and South Carolina do not allow the 1 year holding time. For work performed in these states, the extraction holding time is 7 days for water and 14 days for solid.

[3]Samples collected in 40 mL vials will be extracted by SW846 method 3511 followed by analysis using the LVI procedure described in this SOP.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, *Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and in the Quality Assurance Summaries (QAS) available in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The current MDL value is maintained in the TestAmerica Denver LIMS. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. A batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The minimum batch QC in each run is an acceptable method blank or instrument/calibration blank. See QA Policy DV-QA-003P for further details.

9.4 Method blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water (for aqueous sample batches) or Ottawa sand (for solid sample batches) to which the surrogate compounds are added. The method blank is subject to the entire extraction and analysis process.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (RL) or above one-tenth of the concentration found in the associated samples.

Corrective Action: If the method blank exceeds allowable levels, the source of the contamination must be investigated and all associated samples that produced detections for the contaminant must be re-extracted and reanalyzed. Any samples that produce concentrations more than 10 times the concentration of the same compound as the blank contaminant may be reported with proper flagging and narration.

9.5 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed with each batch of samples. The LCS is

prepared as described in Section 7.8. The LCS is subject to the entire extraction and analysis process.

Acceptance Criteria: The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in LIMS.

Corrective Action: If recoveries are not within the established limits, the analytical system is out of control and corrective action must occur. All associated samples must be re-extracted and reanalyzed. If the LCS exceeds the upper control limit then all samples that do not contain detections for the affected compound may be reportable with client consent and proper flagging and narration.

9.6 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. Preparation of the MS is described in Section 7.9. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS.

Acceptance Criteria: The MS and MSD recoveries must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. The relative percent difference (RPD) between the MS and MSD must be less than the established limit, which is based on statistical analysis of past results, unless otherwise dictated by project requirements. Current control limits are maintained in LIMS.

Corrective Actions: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-extracted and reanalyzed.

If the recovery for any component is outside control limits for both the MS and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-extraction and reanalysis of the batch.

The MS must be analyzed at the same dilution level as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7 Surrogates

Each field sample, QC sample, and each calibration standard that is used for the AR_1660 initial calibration, is spiked with surrogate compounds decachlorobiphenyl (DCB) and trichloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.10.

Acceptance Criteria: The surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise.

Corrective Action: If recoveries of the surrogates in blanks are outside of the control limits, check for calculation or instrument problems. High recoveries might be acceptable if the surrogate recoveries for the samples and other QC in the batch are acceptable. Low surrogate recoveries in the blank require re-extraction and reanalysis of the associated samples especially those that have detections for the targeted compounds that are found in the blank. Samples that are ND may be reportable with proper flagging and NCM.

For field samples, surrogate recovery is calculated and reported for DCB only. TCMX may also be added. However, if both surrogate compounds are added, and recoveries calculated, and either surrogate fails to fall within the control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). Samples with surrogate recoveries that are above the upper control limit may be reportable with flagging and narration if they do not have reportable detections.

If matrix interference is not obvious from the initial analysis, it is only necessary to re-extract and reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effects, as long as the extraction/instrument system is proven to be working properly.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Use the ChemStation chromatography data system to set up GC

conditions for calibration. See Tables 2 and LVI-2 for typical operating conditions. The conditions described in Table LVI-2 are to be used when performing the large volume injection approach.

10.1.2 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.3 Unprocessed calibration data are transferred to the TARGET DB database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration reports are scanned and stored as Adobe Acrobat files on the Public Drive.

10.2 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns.

10.3 Initial Calibration (ICAL)

10.3.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder, *Arizona Calibration Training*.

10.3.2 An external standard calibration using seven concentration levels of the AR_1660 mixture is routinely performed. (At least five calibration levels are required.) This provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

NOTE: See Tables 3 and LVI-3 for Calibration Levels. Calibration levels defined in Table LVI-3 are appropriate when the large volume injection approach is used.

NOTE: Prior to analysis of the initial calibration standards it is recommended that a chlorinated pesticide standard (Method 8081) be analyzed as a locater standard to identify potential interferences in samples due to the presence of chlorinated pesticides.

10.3.3 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.3.4 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds are modeled either as average calibration factors or as calibration curves using a systematic approach to selecting the optimum calibration function.

10.3.5 The calibration for each of the other Aroclors (see Table 1 or LVI-1) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other

commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve for the identified Aroclors. This information is provided to the analyst through special instructions in LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of the preparation of the calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed

10.3.6 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.3.6.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.3.6.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.3.6.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.3.6.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average calibration factors or linear regressions, or six levels for second-order curve fits.

10.3.6.5 If a data point is rejected, it must be documented in the sequence log and on an NCM which is filed with each project that is reported from the calibration.

10.3.7 The high and low standard for the initial calibration of the AR_1660 mixture defines the acceptable quantitation range for all of the Aroclors. The low calibration standard must be at or below the RL. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted and reanalyzed.

10.3.8 Select 5 major peaks in the analyte pattern (only 3 peaks are usable for Aroclor 1221). The peaks that are chosen should have responses that are at least 25% of the response for the largest peak in the Aroclor pattern. Try

to include one peak that is unique (differs in size or location relative to the other common Aroclors) to the Aroclor being quantitated. Calculate the response of each of the major peaks for each Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e., peaks that are significantly larger than would be expected from the rest of the pattern).

NOTE: A minimum of three accurate peaks must be used to quantify an Aroclor (two for Aroclor 1221).

10.4 External Standard Calibration

External standard calibration involves the comparison of instrument responses from the samples to the responses from the target compounds in the calibration standards. The area (or height) of a peak in a sample chromatogram is compared to the area (or height) of the peak in the standard chromatograms that appears at the same retention time. The ratio of the detector response to the concentration of the target analyte in the calibration standard is defined as the calibration factor (CF) and is calculated as follows:

$$CF = \frac{A_s}{C_s}$$ Equation 1

Where:

A_s = Peak area (or height) of the target analyte in the calibration standard.

C_s = Concentration of the target analyte in the calibration standard (µg/mL).

10.5 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met.

10.5.1 Linear Calibration Using Average Calibration Factor

The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

10.5.1.1 The average calibration factor is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ Equation 2

Where:

CF_i = The calibration factor for the i^{th} calibration level.

n = The number of calibration levels.

10.5.1.2 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \text{Equation 3}$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \text{Equation 4}$$

10.5.2 Evaluation of the Average Calibration Factor

Plot the calibration curve using the average CF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., DoD, Arizona and South Carolina require evaluation of each compound individually). Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.5.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b$$ Equation 5

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b$$ Equation 6

Where:

A_s = Peak area (or height) of the target analyte in the calibration standard.

C_s = Concentration of the target analyte in the calibration standard (µg/mL).

10.5.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.99. Some programs (e.g., USACE, AFCEE and DoD) require a correlation coefficient ≥ 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

10.5.5 Second-order regressions and polynomial regression fits of third order or higher are not allowed for this method.

10.6 Second-Source Initial Calibration Verification (ICV)

The second-source ICV standard usually consists of Aroclors 1016 and 1260 only. The stock standards are obtained from a source different than that of the standards used for the calibration. The preparation of the ICV standard is described in Section 7.5. The concentration of each Aroclor in the ICV is 0.25 µg/mL; the concentration of each surrogate is 0.125 µg/mL. The ICV standard is analyzed immediately following the completion of the initial calibration. If any changes are made to the calibration curve types then the ICV must be recalculated to the final form of the ICAL.

If it is necessary to generate a multi-point calibration for any of the other Aroclors, then an ICV standard containing the specific Aroclor(s) is analyzed immediately following the calibration.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% of the expected value. Method 8082A allows a control of ± 20%.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.7 Continuing Calibration Verification (CCV), 0.50 ug/mL.

10.7.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration sequence.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.7.2 It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7). At a minimum, this is analyzed after every 20 samples, including matrix spikes,

LCSs, and method blanks. Some programs, specifically drinking water programs, require a CCV after every 10 samples to minimize the number of samples requiring re-injection when QC limits are exceeded. If 12 hours elapse, analyze the 12-hour standard sequence instead.

10.7.3 RL Standard

It may be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method between every 10 sample injections (see Section 7.7). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit and the samples contain no analytes above the reporting limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria or in the case of matrix effect to confirm the ability to see at the reporting limit.

This procedure is not used when Method 8000C is required.

10.7.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.7.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 15%. Method 8082A requires a control of ± 20%. If a confirmation column is required (see Section 12.5), the CCV criteria must be met on both columns.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of acceptance criteria in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect; if so desired by the client.

Refer to Section 11 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \text{Equation 8}$$

10.7.4.2 Analytes Not Detected (< RL)

For any analyte not detected (ND) in client samples, the %D for that analyte in the bracketing CCVs should also be within acceptance criteria.

However, if the CCV %D exceeds the upper control of the acceptance criteria and the sample results are ND, it still may be possible to report sample results. In this case, the client should be consulted and an NCM written.

If the CCV % D falls below acceptance criteria and sample results are ND, but the target analytes are detected in the RL Standard, it may still be possible to report sample results, since the detection of the analyte(s) in the RL Standard indicate that there was sufficient sensitivity to detect the analyte(s) in the samples. In this case, the client should be consulted and an NCM written. This would only be used in cases where the matrix is affecting CCV recovery and dilution of the affected sample(s) is not an alternative.

NOTE: The state of Arizona requires the use of Method 8000C and does not allow the use of the average %D.

10.8 Retention Time (RT) Windows

10.8.1 Determine the retention time (RT) windows for the 5 major peaks selected for each Aroclor (3 peaks for Aroclor 1221). The AR1016 windows will be used to establish retention time windows for AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for AR1254, AR1260, AR1262, and AR1268.

10.8.2 Determine new RT windows each time a new column is installed or annually.

10.8.3 Inject a standard containing all analytes at least once each day over a 72-hour period.

10.8.4 Calculate the mean and standard deviation of the three RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \quad \text{and} \quad SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RT_i - \overline{RT}\right)^2}{n-1}} \qquad \text{Equations 9 and 10}$$

Where:

RT_i = Retention time for the i^{th} injection.
n = Number of injections (typically 3).
SD = Standard deviation.

10.8.5 The width of the RT window for each analyte is set at ± 3 times the standard deviation of the RTs determined for each analyte over the 72-hour period. For multi-response analytes, use the RT of major peaks.

10.8.6 The center of the RT window for each analyte is the RT from the last of the three analyses of the standard.

10.8.7 The center of the window for each analyte is updated with the RT from the last of the three standards measured for the 72 hour RT study, the level 4 standard of the ICAL, or the CCV at the beginning of the analytical sequence. The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure.

10.8.8 If the width of the RT window, as calculated above, is less than ±0.03 minute, use ± 0.03 minute as the RT window width. This allows for slight variations in RTs caused by sample matrix.

Acceptance Criteria: The RT for each compound in each CCV analysis must be within the RT windows established by the daily initial CCV.

Corrective Action: If a target analyte falls outside the established RT window in a CCV standard, either adjust the center of the window based on the CCV, or investigate the problem and calculate new RT windows. All samples analyzed after the last acceptable CCV must be reanalyzed.

10.8.9 Sample Retention Time Criteria

The surrogate must fall within the established RT window. Target analyte peaks must be within the established RT window to be reported as such. If the surrogate RT indicates a RT shift, it may be possible to accept a target analyte peak if it has not shifted relative to the surrogate peak. The presence of a definitive aroclor pattern will be positive evidence of a hit and may supersede RT window criteria. An NCM should be written to explain this case.

10.8.10 Daily Retention Time Windows

The centers of the retention time windows are adjusted at the beginning of each analytical sequence based on the daily initial CCV.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed

must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be checked for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 The final extract volume in hexane is 10 mL.

11.3.5 Use hexane to dilute sample extracts, if necessary.

11.4 Gas Chromatography

Chromatographic conditions for this method are presented in Tables 2 and LVI-2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the TARGET DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the TARGET DB software.

11.5 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. For LVI analysis 10 µL of sample extract is introduced into the chromatographic system. Samples, standards, and QC samples must be introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.6 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Tables 3 and LVI-3 for the calibration levels used.

11.6.1 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.7.1) and updating the retention time windows (Section 10.8.7)

11.6.2 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration

verification. Any samples that were not bracketed by a closing CCV must be reanalyzed in the new 12 hour sequence.

11.6.3 The following is a typical analytical sequence for routine sample analysis:

- Primer (Injection of any standard that contains any of the analytes to establish the stability of the chromatographic system.)
- Hexane instrument blank
- Daily initial CCV (Unless an ICAL is performed, which is immediately followed by the second-source initial calibration verification.)
- 10 sample injections (The first set of samples analyzed usually includes the method blank and the LCS, and may include matrix spikes.)
- CCV
- Followed by cycles of 10 sample injections and a CCV, as needed
- Closing CCV, instrument blank, and RL Standard

11.7 Retention Times

The centers of the RT windows determined in section 10.8 are adjusted to the RT of each individual peak as determined in the 12-hour calibration verification. The RT window must be updated at the beginning of each analytical sequence.

11.8 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

11.9 Upon completion of the analytical sequence, transfer the raw chromatography data to the TARGET DB database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See DV-QA-011P for requirements for manual integration. Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.10 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.10.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.10.2 Perform a level 1 data review and document the review on the data review

checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.10.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the annual integration summary report and/or the level 2 review checklist. For Federal projects and certain client specified projects, the documentation of the manual integration review must be scanned and attached to the project tin the LIMS to be included with the Level 4 data package. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12 Calculations / Data Reduction

12.1 Detailed equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and under the public folder, Arizona Calibration Training.

12.2 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 5 major peaks (3 peaks for Aroclor 1221). The peaks must be within the established retention time windows. If there is an interference that affects the accuracy of results, the analyst may use as few as 3 major peaks (2 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation.

12.4 Second column confirmation of Aroclors is performed only when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: USACE and DoD projects require the use of second-column confirmation of Aroclors unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

NOTE: South Carolina requires second column confirmation.

NOTE: DOE requires second column confirmation with flagging if the results vary by more than 25% RPD.

NOTE: Method 8082A indicates that second column confirmation is necessary when the sample composition is not well characterized.

12.5 Dual Column Quantitation

NOTE: Dual column quantitation is not routinely performed for PCB analysis. This section is included for those clients/projects that require dual column confirmation.

12.5.1 A primary column is designated. If the continuing calibration fails for one of the columns then the appropriate corrective action must be taken. The result from the primary column is normally reported. The result from the secondary (confirmatory) column is reported if any of the following is true:

12.5.1.1 There is obvious chromatographic interference on the primary column.

12.5.1.2 The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident on the primary column.

12.5.2 Dual Column Results With > 40% RPD

12.5.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.5.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.5.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.5.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{\left|R_1 - R_2\right|}{\frac{1}{2}\left(R_1 + R_2\right)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.5.3 If total Aroclors and dual column quantitation is requested, then total aroclors will be calculated for each column by summing the detections on each column. If the results for total aroclors from the primary column differs from the total aroclor result from the secondary column by more than 40%, the total aroclors result from the primary column will be reported and the data will be flagged accordingly.

12.6 Surrogate Recovery

12.6.1 Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB).

12.6.2 In cases where the addition of the surrogate tetrachloro-*m*-xylene (TCMX) is required, its recovery is calculated and reported. In cases where both surrogates are added and recoveries calculated, the recovery of each surrogate is evaluated and corrective action must be taken if either surrogate recovers outside of the established control limits and matrix interference is not evident. Depending on project requirements, corrective action may be necessary only if DCB and TCMX are both outside of acceptance limits.

12.7 Calibration Range and Sample Dilutions

12.7.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when blanks or other samples were analyzed after a sample with similar high concentration or when the detection in the sample with suspected carryover is much higher than the expected amount of the carryover (i.e., the suspect sample's concentration is similar to or higher that the sample run previous to it). It may also be necessary to dilute samples because of matrix interferences.

12.7.2 If the initial diluted run has no hits or hits below 20% if the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.7.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.7.4 Reporting Dilutions

Some programs (e.g., South Carolina, DoD, and AFCEE) and some projects require reporting of multiple dilutions (check Method Comments in

LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.8 Interferences are Observed in Samples

12.8.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.8.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.8.3 Suspected Positive Interferences

If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option. At a minimum, the Data Review Template prepared by the analyst should include the following comment for inclusion in the case narrative:

"Based on review of the chromatograms for samples _____, it is my opinion that the evident interferences may be causing false results.

Date _____ Analyst _____"

12.9 Identifying and Reporting PCBs

12.9.1 In samples where the PCB pattern matches an individual Aroclor reasonably well, the samples should be quantified and reported as usual. When there are numerous PCB peaks present but there are no good matches to any individual Aroclor, choose the Aroclor (or Aroclors) that most closely match the sample and quantify the peaks as that Aroclor. The sample should not be reported as "not detect" based solely on the absence of a good match to a single Aroclor mixture. Multiple Aroclors should only be reported if their patterns are reasonably well separated. For example, 1232 and 1254 could be reported together, but not 1242 and 1248. See Attachment 1 for additional information on identifying Arochlors.

NOTE: When reporting and quantifying PCBs that do not closely match an Aroclor standard, it is absolutely essential and mandatory that this is explained in the report narrative.

12.9.2 Some example text that can be used in the report narrative is presented below:

Sample XXXX appears to contain PCBs based on the presence of numerous PCB peaks. However, due to weathering or other environmental processes, the PCBs in the sample do not closely match any of the Aroclor standards we use to calibrate our instruments. We quantified and reported the sample as Aroclor ZZZZ (or as a mixture of Aroclors ZZZZ and YYYY). Due to the poor match with the Aroclor standard(s), there is increased qualitative and quantitative uncertainty associated with this result. This approach is consistent with the guidance in section 7.9.3 of SW846 method 8082A. If these results do not meet the needs of your project then we would suggest a further analysis of the sample. Depending on the objectives, this may include congener-specific analysis by 8082A; or analysis a more specific method (e.g., method 1668 or an adaptation of method 8270) for PCB congeners or PCB homolog totals.

Some clients may insist on ND reporting if the patterns are not clear. In that event, we should add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, we would still insist on a narrative as suggested in the previous paragraph.

12.9.3 Sample Matrix Issues

12.9.3.1 In some cases when analyzing for multi-component analytes, the sample matrix is so complex that it would obliterate any possible pattern that would allow us to identify the analyte. When this happens, it is true that the analyte is not detected at the normal detection limit. However, it is true that we could not have detected the analyte at the normal detection limit. Even if the analyte was present, we would not be able to recognize it.

12.9.3.2 When this occurs, the sample must be analyzed at a dilution that would allow us to detect the analyte, and the reporting limit should be the one appropriate for that dilution. Reporting a non-detect at the normal reporting limit is not an acceptable practice.

12.9.3.3 Some clients may insist on ND reporting if the patterns are not clear. In that event, add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, a narrative comment as suggested in the previous paragraph is still required.

12.9.4 Background on PCBs

12.9.4.1 PCBs were widely used in a variety of products prior to being banned in the 1970's. The most common usages were in electric motors and transformers. They were manufactured by gas phase chlorination of a biphenyl molecule. The nomenclature, in general, describes the weight percent of chlorine in the final product. Thus, Aroclor 1254 was produced by chlorinating a quantity of biphenyl until the resulting product was 54% chlorine by weight. Aroclor 1242 was 42% chlorine by weight.

12.9.4.2 PCBs were manufactured in batch processes, so there were slight variations between batches, but in general each Aroclor had a very reproducible pattern of chlorinated biphenyl isomers (congeners). With few exceptions, when we detect PCBs in the environment the initial contaminant was one of the Aroclors.

12.9.4.3 The one exception to the nomenclature of the Aroclors is Aroclor 1016. In the 1960's researchers started to find PCBs in fish tissue in the Great Lakes. The primary congeners appearing in the fish were pentachlorobiphenyls. The manufactures of PCBs devised a synthetic process that created an Aroclor with very similar properties to Aroclor 1242, but minimized the formation of pentachlorobiphenyl molecule. Aroclor 1016 41% chlorine by weight and as result it can be difficult to distinguish from 1242.

12.9.4.4 While the pattern of congeners was quite reproducible in the pure products once in the environment the pattern changes. The lesser chlorinated PCBs are more water soluble and are more volatile, while the more highly chlorinated PCBs bind to solids and sediments more strongly. As examples, landfill gas condensates tend to have a bias toward the lesser chlorinated congeners because they are more volatile. River sediments near source of PCBs tend to have a bias toward the more highly chlorinated congeners because the accompanying lesser chlorinated congeners were more water soluble. Downstream from the source of contamination, however, there will be a bias toward the less chlorinated congeners because the more heavily chlorinated congeners were trapped in the sediments near the outfall. Anaerobic and aerobic microbial degradation reduce the concentrations of some congeners and an increase in concentrations of others. Although they are rarely the primary mechanisms, oxidative and photolytic processes are also selective, impacting some congeners more than others.

12.9.4.5 As a result, PCBs in the environment rarely have an exact match to the Aroclor standards that we use to calibrate our instruments. There is inevitably some level of judgment required to choose the Aroclor that has the best match to the sample in questions. Sometimes this is straightforward, but other times the judgment is difficult and can be controversial. In the worst cases, we can

have situations where there are clearly PCB peaks throughout a chromatogram, but there is no good match with any of the Aroclors. It is recommended that at a minimum there must be some peak groupings present that are characteristic of an aroclor pattern in order to indicate a positive detection.

12.10 Calculations

12.10.1 Concentration of Analyte in Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.5 for details on establishing the calibration function):

12.10.1.1 Average Calibration Factor:

$$C_e = \frac{A_e}{\overline{CF}} \qquad \text{Equation 12}$$

12.10.1.2 Linear Regression:

$$C_e = \frac{\left[A_e - b\right]}{a} \qquad \text{Equation 13}$$

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_e = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

12.10.2 Concentration of Analyte in Original Sample

The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\ \frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \text{Equation 14}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).
DF = Dilution Factor (post extraction dilutions)

12.10.3 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.10.4 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA} \right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.
SR = Measured concentration in unspiked sample.
SA = Concentration of spike added to sample.

12.10.5 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{\left| R_1 - R_2 \right|}{\frac{1}{2}\left(R_1 + R_2 \right)} \times 100\% \qquad \textit{Equation 17}$$

Where *R1* is the result for the MS and *R2* is the result for the MSD.

12.11 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy DV-QA-005P. Each of the other aroclors have an MDLV performed annually to satisfy NELAC 2003 requirement. For DoD, AFCEE, DOE and Texas TRRP projects, AR_1660 MDLVs and LOQVs are performed quarterly. MDLs and LOQs are stored in LIMS.

13.2 Demonstration of Capabilities

An initial demonstration of capability for each method must be performed prior to analyzing samples in accordance with DV-QA-0024.

13.2.1 For the standard analyte list, the initial demonstration consists of the preparation and analysis of a QC check sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described in Section 12.1).

13.2.2 Four aliquots of the QC check sample are analyzed with the same procedures used to analyze samples, including sample preparation.

13.2.3 The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples.

13.2.4 For non-standard analytes an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is successful analysis of an extracted standard at the reporting limit and a single point calibration.

13.3 Training Requirements

13.3.1 The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.3.2 Each analyst performing the method must complete an initial demonstration of capability (IDOC) by successfully preparing and/or analyzing four consecutive LCSs, or a blind performance evaluation (PE) sample, or other acceptable QC samples. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files. Analysts who continue to perform the method must successfully complete a demonstration of capability annually.

14 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention.

14.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Waste hexane solvent: Flammable Solvent – Waste Stream C

15.2.2 Vials containing extracts in hexane: Expired Extract Vials – Waste Stream A

15.2.3 Concentrated sulfuric acid and hexane from sample cleanup: Concentrated Acids with Organics - Waste Stream V

15.2.4 Expired reagents and standards – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.2.5 Samples containing polychlorinated biphenyls (PCB's) at concentrations ≥50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Waste Coordinator immediately if a sample falls into the TSCA category.

16 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.4 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.1.5 Method 3580A, Waste Dilution, Revision 1, July 1992.

16.1.6 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.7 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.1.8 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December, 1996.

16.1.9 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 1, February 2007

16.1.10 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.11 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

17 Method Modifications

Item	Method	Modification
1	8082	Method 8082 includes an internal standardization option. Because of the high probability of interferences affecting internal standards, this is strictly an external standard SOP.
2	8000B	Method 8000 allows for use of a second order or third order calibration curve. TestAmerica Denver does not allow for any curvilinear calibrations for the analysis of arochlors.

18 Attachments

Table 1: Analyte List and Standard Reporting Limits

Table 2: Typical Instrument Conditions

Table 3: Calibration Levels (µg/mL)

Table 1-LVI: Analyte List and Standard Reporting Limits using Large Volume Injection

Table 2-LVI: Typical Instrument Conditions using Large Volume Injection

Table 3-LVI: Calibration Levels (µg/mL) using Large Volume Injection

Attachment 1: Arochlor Identification 101

19 Revision History

Revision 6, June 15, 2012

- Added Tables 1-LVI, 2-LVI, and 3-LVI for large volume injection

Revision 5.1, January 16, 2012

- Changed extraction holding time for water and solid to 1 year with exclusion for California, Connecticut, Pennsylvania and South Carolina (Section 8).
- Reformatted paragraphs throughout

Revision 5, December 2011

- Combined SOP DV-GC-0021 and DV-GC-0030 Rev. 0.2. Upon implementation of this revision of SOP DV-GC-0021, SOP DV-GC-0030 will be deactivated.

- Added details for analysis of polychlorinated terphenyls by this procedure (sections 1, 3, 7 and Table 1).
- Updated Section 6 to include reference to master list of documents, software and hardware and volumetric flasks.
- Updated refrigerator temperature references from 4 $\pm$ 2°C to 0-6°C throughout.
- Updated vendors and catalog numbers for standards (Section 7)
- Updated Section 9 for consistency with SOP DV-QA-003P.
- Added calibration section to describe calibration models.
- Revised Procedure (new section 11) to be consistent with other SOPs revised in the last year.
- Added detail about review process (Section 11.8)
- Revised Calculations section (new section 12) to address dual column quantitation, sample dilution, and recovery calculations.
- Revised section numbers for previous sections 12-18.
- Updated Method Modifications section
- Revised Table 1 and Table 2

Revision 4.1, December 2010

- Added QC criteria for cleanup procedures to section 4.1.
- Added section 11.1 to reference corporate SOP CA-Q-S-005 “Calibration Curves”

Revision 4.0, June 2010

- Annual Technical Review.
- Deleted the centering of the window requirement for “each subsequent 12-hour calibration verification” in Section 9.19.
- Added LOQV information in Section 12.1
- Added Attachment 1

Revision 3.1, June 2009

- Basic Annual Review

Revision 3, April 2008 updated to TestAmerica and reformatted.

- Updated formatting to comply with Policy DV-QA-001P.
- Added Section 1.3 to reference sample preparation SOPs..
- Added references to sample preparation SOPs to Section 2.
- In Section 3.2, added note to explain the existence of anomalous formulations of Aroclors.
- Updated information on standards in Section 7 to reflect current practice.

- Revised Section 9.1 to include reference to Policy DV-QA-024P for QC requirements for federal programs.
- In Section 10, deleted instructions for second-order calibration curves, which are not used for this method.
- Added the RL Standard to Sections 7.6 and 10.7.3.
- Added information for setting up and running specific laboratory instrumentation in section 10.1, 11.5, 11.6, and 11.10.
- Updated data analysis and calculations in Section 12 to reflect current practice.
- Updated Sections 13, 14, and 15 to reflect current practice.
- Expanded the references in Section 16.

Table 1. Analyte List and Standard Reporting Limits

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	1.0	33
Aroclor 1221	1.0	47
Aroclor 1232	1.0	33
Aroclor 1242	1.0	33
Aroclor 1248	1.0	33
Aroclor 1254	1.0	33
Aroclor 1260	1.0	33
Aroclor 1262	1.0	33
Aroclor 1268	1.0	33
PCT 5432	0.5	50
PCT 5442	0.5	75
PCT 5460	0.5	50

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions
Injection Port Temperature:	250 ^{o}C
Detector Temperature:	325 ^{o}C
Temperature Program:	Instrument W 125 ^{o}C for 1 minute 8 ^{o}C/min to 275 ^{o}C for 0.1 minute 30 ^{o}C/min to 310 ^{o}C for 2 minutes Instrument P3 125 ^{o}C for 1.25 minutes 30 ^{o}C/min to 180 ^{o}C 12 ^{o}C/min to 280 ^{o}C 15 ^{o}C/min to 320 ^{o}C for 2.6 minutes
Column 1:	CLPI, 30 m x 0.32 mm id, 0.5 µm
Column 2:	CLPII, 30 m x 0.32 mm id, 0.25 µm
Injection:	1 or 2 µL
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek or J&W or Supelco glass tee, single gooseneck liner

Table 3. Calibration Levels (µg/mL)

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1221	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1232	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1242	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1248	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1254	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1260	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1262	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1268	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05
Decachlorobiphenyl	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05

Table 1-LVI. Analyte List and Standard Reporting Limits for Large Volume Injection

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	0.5	5.0
Aroclor 1221	0.5	5.0
Aroclor 1232	0.5	5.0
Aroclor 1242	0.5	5.0
Aroclor 1248	0.5	5.0
Aroclor 1254	0.5	5.0
Aroclor 1260	0.5	5.0
Aroclor 1262	0.5	5.0
Aroclor 1268	0.5	5.0
PCT 5432	NA	NA
PCT 5442	NA	NA
PCT 5460	NA	NA

Table 2-LVI. Typical Instrument Conditions for Large Volume Injection

Parameter	Recommended Conditions
Injection Port Temperature:	250 ^{o}C
Detector Temperature:	325 ^{o}C
Temperature Program:	Instrument P3 125 ^{o}C for 1.25 minutes 30 ^{o}C/min to 180 ^{o}C 12 ^{o}C/min to 280 ^{o}C 15 ^{o}C/min to 320 ^{o}C for 2.6 minutes
Column 1:	Restek Rtx-CLPesticides 30m X 0.32 mm id, 0.5 µm (Cat# 11139 or equivalent)
Column 2:	Restek Rtx-CLPesticides2 30m X 0.32 mm id, 0.5 µm(Cat# 11324 or equivalent)
Injection Volume:	10 µL (Agilent 25 µL G4513-80241 or equivalent)
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek Universal Presstight Connector (Cat# 20400 or equivalent)
Injection Port Liner:	Agilent 5190-2293 90011 or equivalent

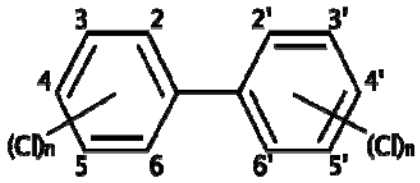
Table 3-LVI. Calibration Levels (µg/mL) for Large Volume Injection

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1221	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1232	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1242	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1248	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1254	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1260	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1262	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Aroclor 1268	0.0025	0.005	0.01	0.025	0.05	0.075	0.10
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.000125	0.00025	0.0005	0.00125	0.0025	0.00375	0.005
Decachlorobiphenyl	0.000125	0.00025	0.0005	0.00125	0.0025	0.00375	0.005

Attachment 1. Arochlor Identification 101

Aroclor identification 101

It can be difficult to correctly identify which Aroclor is present in a sample. This document provides a few guidelines. We are calling this document Aroclor identification 101 not because it is simple, but because Aroclor identification 201 and 301 (mixed, weathered Aroclors) are much more difficult still (sort of like P-Chem!)



First, we should consider what Aroclors actually are: They are mixtures of polychlorinated biphenyls.

Each phenyl ring can accommodate between zero and 5 chlorines. There are 209 possible isomers with 1-10 chlorines (the surrogate decachlorobiphenyl is the fully chlorinated molecule). Of these, about 130 are present in various Aroclor mixes, accounting for the complexity of the chromatograms. The first two digits of the Aroclor number refers to the number of carbon atoms, the last two refer to the degree of chlorination. Thus Aroclor 1248 has 12 carbon atoms in each molecule, and 48% chlorine by mass. So, as the last two digits increase, the overall degree of chlorination increases, the volatility decreases, and the pattern of peaks moves later in the chromatogram. Arochlor 1248 consists of approximately 1% monochlorobiphenyl, 13% dichlorobiphenyl, 45% trichlorobiphenyl, 31% tetrachlorobiphenyl and 10% pentachlorobiphenyl.

It is estimated that 1.25 billion pounds of PCBs were produced until Monsanto ceased production in 1977. PCBs are very persistent, so much of this material is still present in the environment.

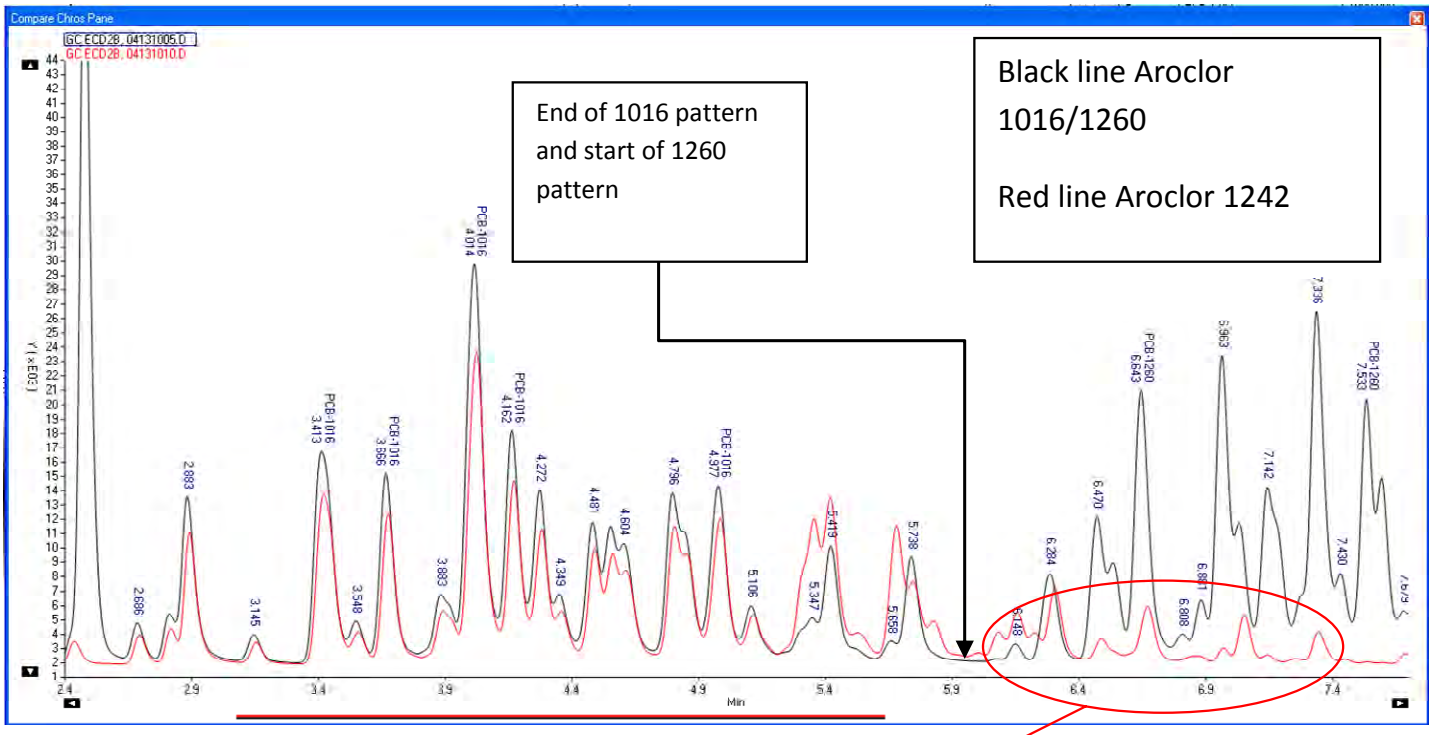
Example Chromatograms

In the following examples I'll refer to retention times frequently – your retention times will of course be different because of different chromatographic conditions but the same principles apply.

Attachment 1. Arochlor Identification 101 (Continued)

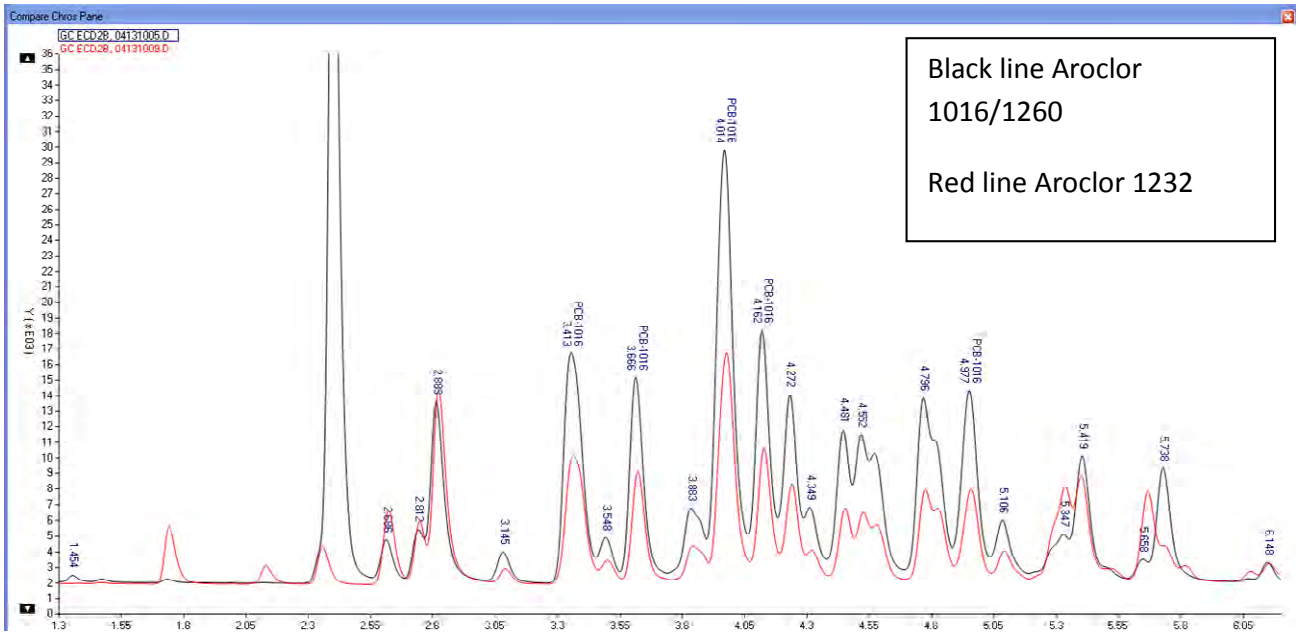
The first example considers Aroclor 1016 vs. 1242. Note that the early part of the chromatograms (2.5 – 5.3 minutes) are virtually identical. The key difference is the presence of some later eluting peaks (6.2-7.3 minutes in this chromatogram) in 1242 that are not present in 1016. This difference is masked by the fact that Aroclor 1260 is also present in this standard. Most labs analyze standards of 1016 and 1260 together – there is nothing wrong with this but it is a good idea to periodically (one run with each initial calibration?) analyze them separately so that you have a good idea of the two separate patterns.

The story of 1016 is interesting – in the early 1970's PCBs were starting to be found in fish in the Great Lakes. The more heavily chlorinated biphenyls were bioaccumulating more and were of greatest concern. So, Monsanto attempted to modify the manufacturing process to reduce the amount of pentachlorobiphenyls in Aroclor 1242, while still keeping the overall degree of chlorination similar. They were successful in this regard – Aroclor 1242 has about 10% pentachlorobiphenyls which show up between 6 and 7.4 minutes in the chromatogram below. Aroclor 1016 has 42% by weight chlorine (it does not follow the standard naming convention) but has no pentachlorobiphenyls.

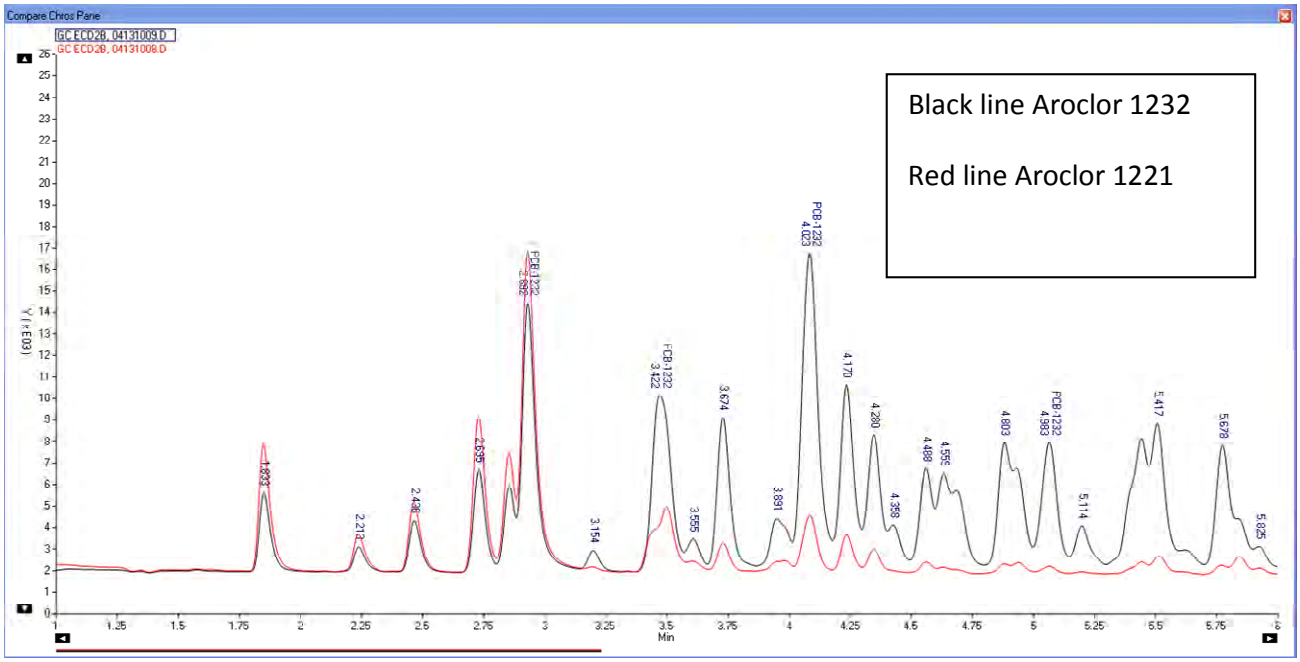


Attachment 1. Arochlor Identification 101 (Continued)

Here is 1016 vs. 1232. These are even more similar (the large peak at around 2.35 min is TCMX) but note the very early peaks present in 1232 and not in 1016, and also note that the front end is stronger in 1232 for example in 1232 the peak at 2.88 min is about the same size as those at 4.79 and 4.97, whereas in 1016 the later peaks are twice as large.

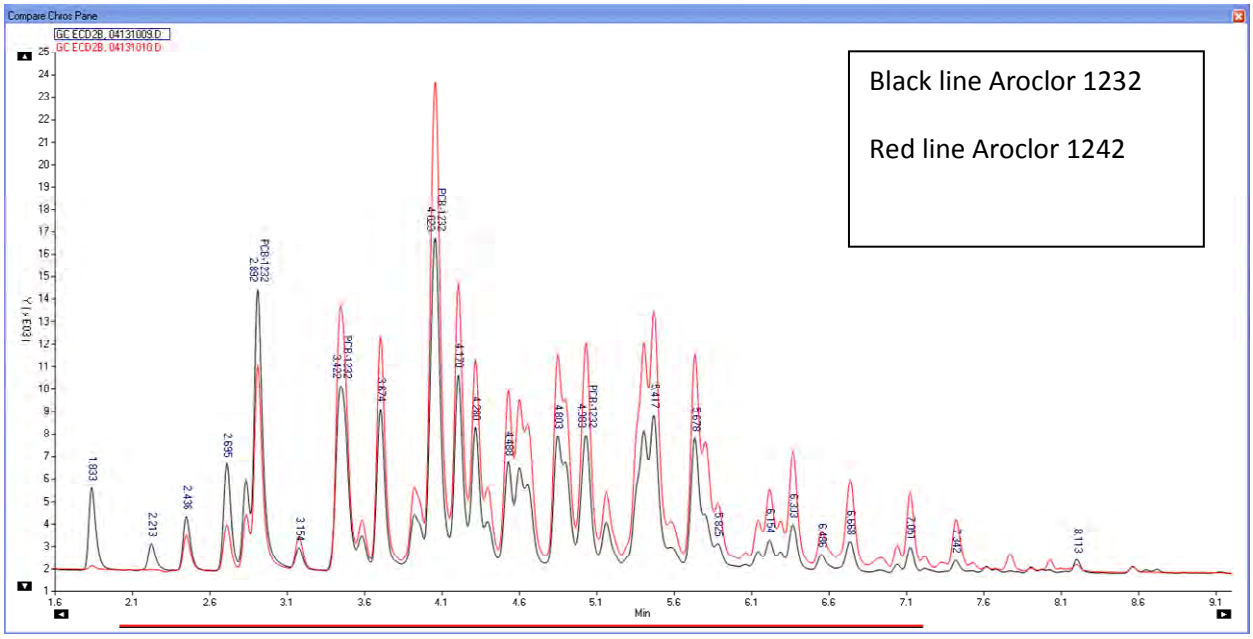


For 1221 vs. 1232, the front end of the chromatogram is identical, but 1232 has later peaks that are not present in 1221.

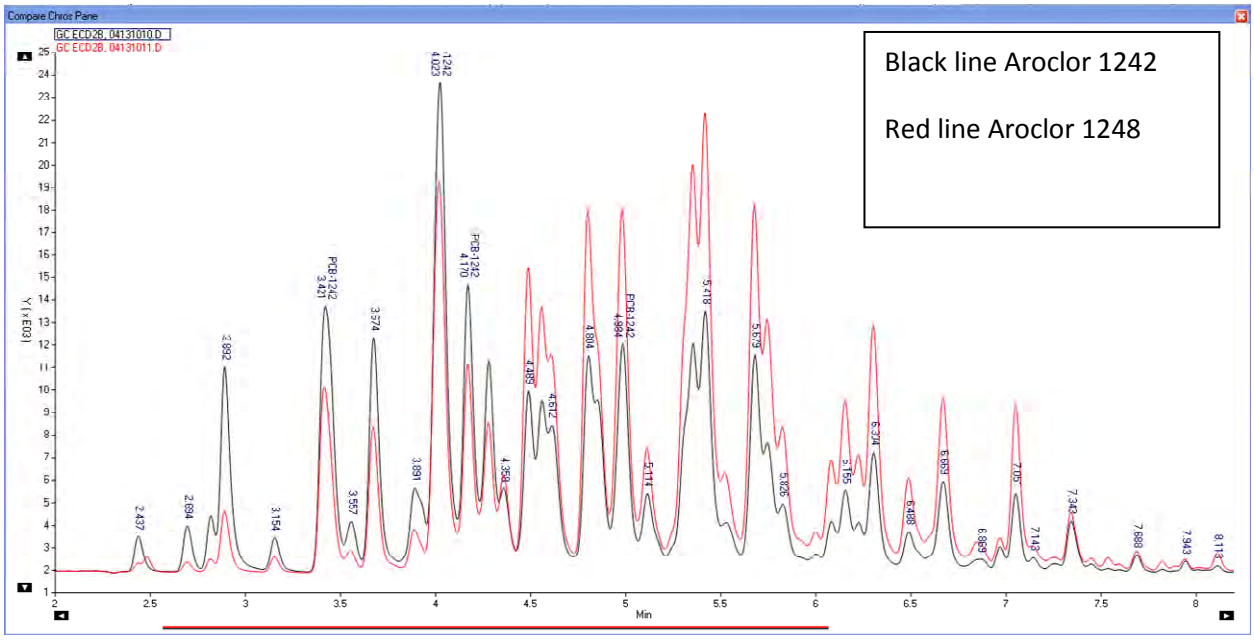


Attachment 1. Arochlor Identification 101 (Continued)

1232 and 1242 are best distinguished by the early peaks in 1232 (1.83, 2.13) that are not present in 1242. Also note that the peak at 2.89 is twice the height of that at 5.41 in 1232, whereas the 5.41 peak is slightly higher in 1242. This relative size of the front and back end of the envelope is a key tool for distinguishing Aroclors.

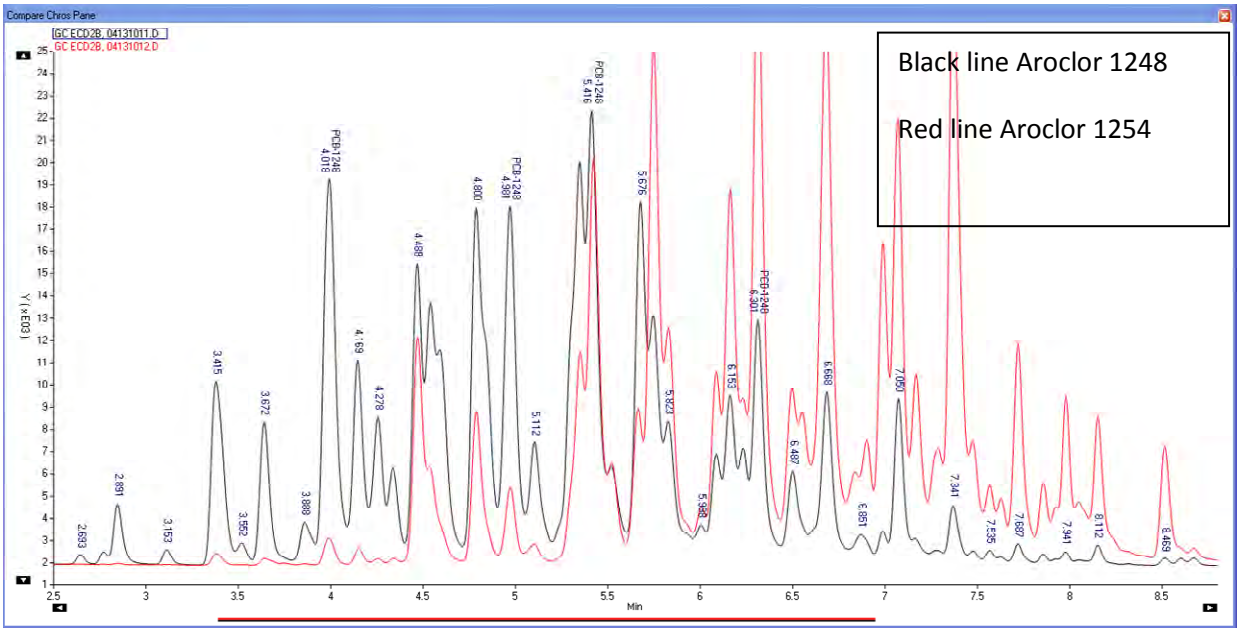


Aroclors 1242 and 1248 both have all of the same peaks, so the relative strength of the front and back of the envelope is the only way to distinguish. For example, in 1242, the peak at 3.42 is larger than that at 5.67, whereas for 1248, the 5.67 peak is considerably larger than the 3.42 peak.

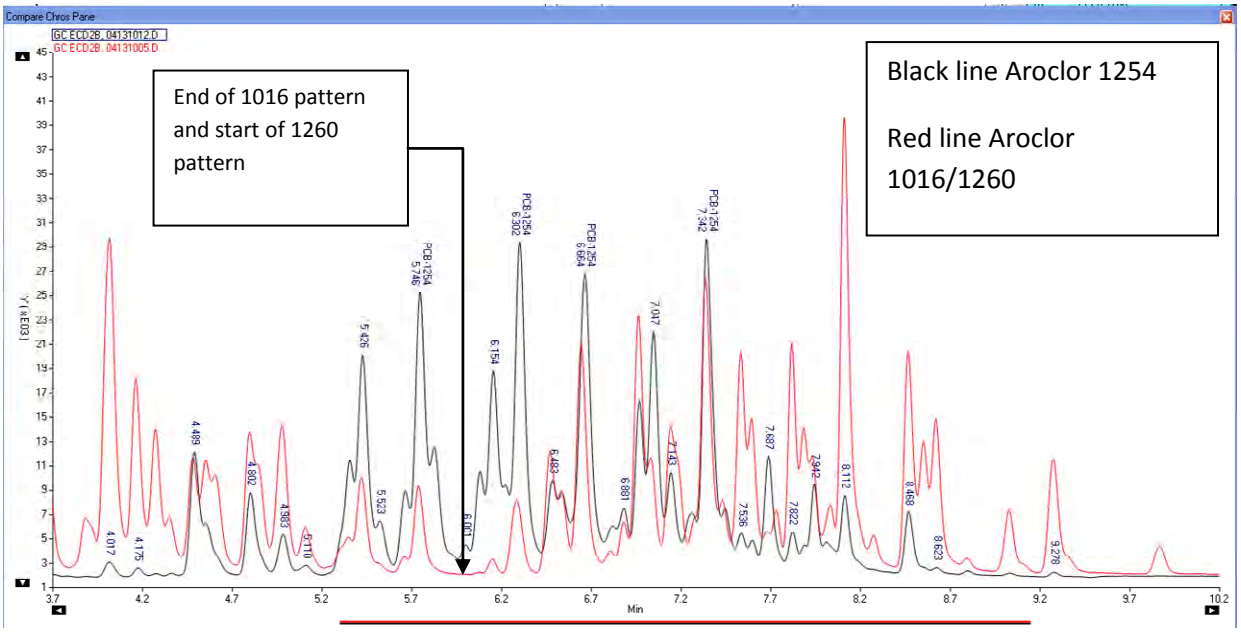


Attachment 1. Arochlor Identification 101 (Continued)

1248 vs 1254 is a relatively easy case, the front end of the envelope is much stronger in 1248.

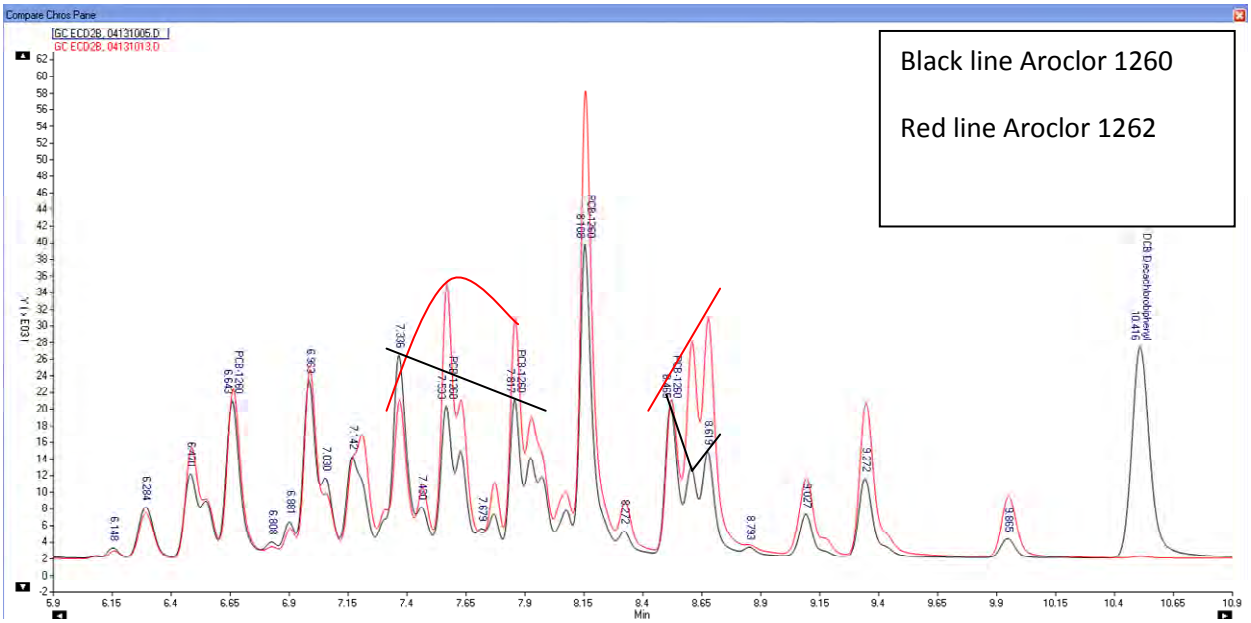


The 1254 vs. 1260 chromatograms are again a little masked by the inclusion of 1016 in the 1260 standard (peaks up to 5.9 min in the 1260 chromatogram actually belong to 1016). Keeping this in mind, the presence of peaks at 5.42 and 5.74 indicates 1254. The relative strength of peaks in the 7.5-9.3 range indicates 1260.

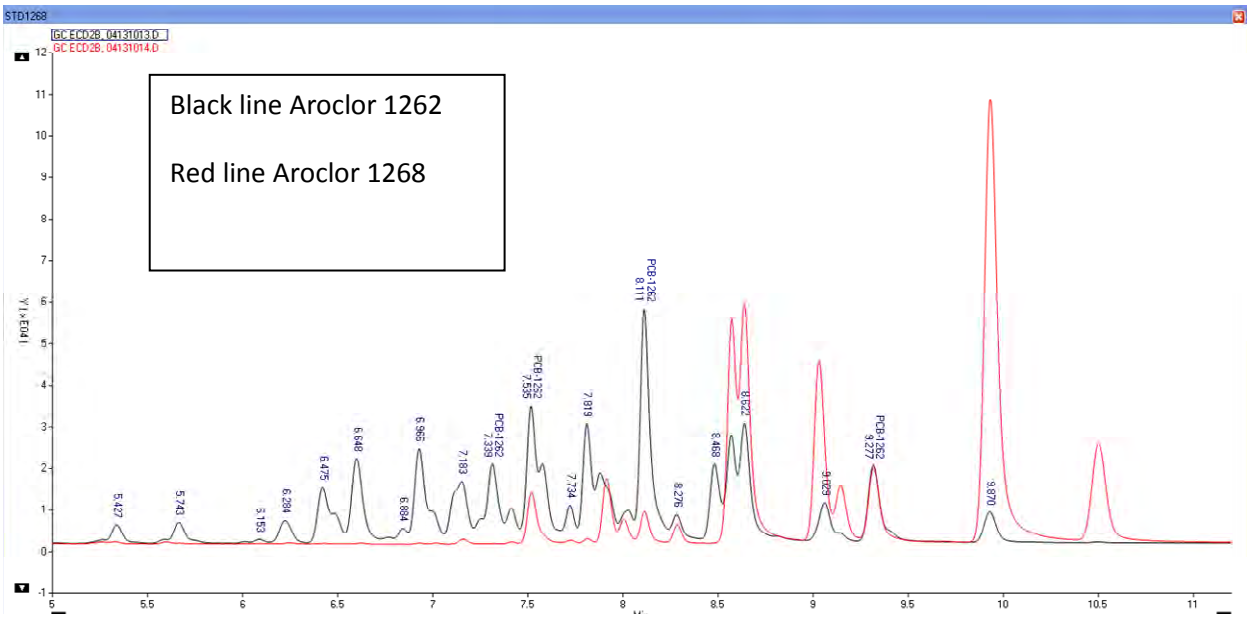


Attachment 1. Arochlor Identification 101 (Continued)

Many labs do not analyze for 1262, perhaps just as well since it is certainly challenging to distinguish from 1260. However, the shape of the envelope is again the key. Note that in 1262 the peaks around 8.6 minutes are as large as that at 6.96, whereas they are only half the size in 1260. Also note the shape of the envelope for the peaks in the 7-8 minute range – bow shaped for 1262 and a straight declining line for 1260. The envelope shape is also quite different in the 8.4-8.7minute range.

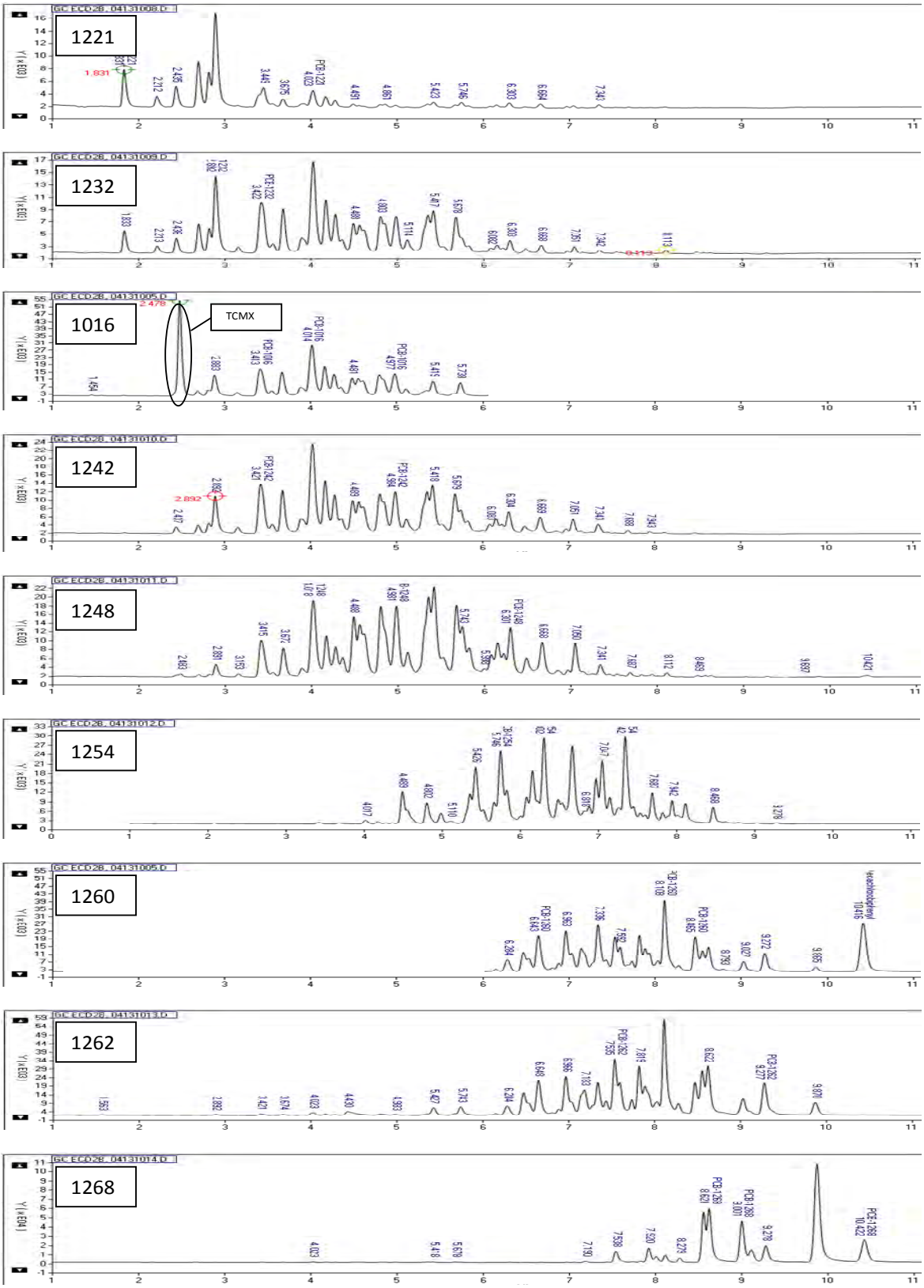


The really strong peak at 9.87 and the lack of much of a pattern between 6.0 and 7.5 minutes are good indicators of 1268.



Attachment 1. Arochlor Identification 101 (Continued)

AROCLOR PATTERNS



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Title: Microwave Extraction of Solid Samples by Method [SW-846 3546]

Approvals (Signature/Date):

Susan H. Decker 1/6/11
Susan Decker — Date
Technical Manager

Adam W Alban 10 Jan 11
Adam Alban — Date
Health & Safety Manager / Coordinator

1/11/11
John Morris — Date
Quality Assurance Manager

Robert C Hanisch 1/11/11
Robert C. Hanisch — Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing the sample and organic solvent. This procedure achieves analyte recoveries equivalent to those from soxhlet or sonications methods, but uses less solvent. This SOP is based on SW-846 Method 3546.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is solvent extracted using a microwave extractor.

3.0 Definitions

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Sodium sulfate should not be used in the extraction vessel. Salts are known to super heat when exposed to microwave energy. Samples are extracted without the addition of sodium sulfate, but the extracts are dried with sodium sulfate after the extraction, before concentration of the extracts.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 A post-run cool down must be used after each extraction to prevent the possibility of operator burns. Pressure builds up in the closed vessel at high temperatures. Care should be taken when opening the vessel when it is above room temperature.

5.1.2 Eye protection that satisfies ANSI Z87.1 (as described in the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in**

the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous. It is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

- Microwave extractor. CEM MARS®
- Microwave extraction vessels. 75mL Teflon™ Express vessels with stopper and cap (CEM Corp.)

- Hand wrench to tighten the caps on the extraction vessels.
- MARS 40 position carrousel (CEM Corp)
- Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.
- Media bottles, 100 mL with Teflon™-lined caps.
- Stainless steel conical funnels
- Ashless cellulose filter paper – Whatman Grade 41 or Ahlstrom Grade 54
- Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.
- Metal spatulas or tongue depressors.
- Solvent dispenser pump.
- Filter flask.
- Vacuum pump.
- Washing tool for Teflon™ extractor vessels. This tool is a long thin sponge-like brush.

6.1 Computer Software and Hardware

- Please refer to the master list of documents and software located on G\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Methylene chloride – Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.2 Acetone - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Hexane - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Baked Sodium Sulfate, 12-60 mesh - Heat sodium sulfate in a 400 °C oven for at least four hours. Each lot is tested following CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400 °C oven for at least four hours.

7.6 35% Nitric Acid – Dilute 70% Nitric Acid 1:1 in water.

Standards

7.7 Please reference SOP DV-OP-00020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass with Teflon-lined lids	30 grams	Cool 4 $\pm$ 2°C	14 days	40 CFR Part 136.3
Wipes	Glass with Teflon-lined lids	N/A	Cool 4 $\pm$ 2°C	14 days	40 CFR Part 136.3

[1] Exclusive of analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, Method Comments and QAS to determine specific QC requirements that apply.

The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in DV-QA-003P, Quality Assurance Program.

Specific QC requirements for Federal programs, e.g., Department of Defense (DoD) Department of Energy (DoE), AFCEE etc., are desribed in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each

analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

The method blank consists of 30g of baked Ottawa sand free of any of the analyte(s) of interest.

Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 10% of the analyte concentration found in the associated samples, whichever is higher. Note that some programs (e.g., AFCEE, Navy, and USACE) require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: If target analytes in the blank exceed the acceptance limits, an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.5 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

The LCS consists of 30g of baked Ottawa sand to which the analyte(s) of interest are added at known concentration.

Method AK102 requires LCS and a LCSD for every batch for every spike compound.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of

historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

Corrective Action: If analyte recovery or RPD falls outside the acceptance range, but the associated LCS recovery is in control, and all other QC criteria (e.g., continuing calibration verification) are met, then there is no evidence of analytical problems, and qualified results may be reported. The situation must be described in an NCM and in the final report case narrative. In other circumstances, the batch must be re-prepared and reanalyzed.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Critical Procedural Considerations

10.2.1 As stated throughout this SOP, analysts must review the LIMS Method Comments, and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-009).

10.2.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.3 Periodic acid cleaning.

10.3.1 Once a week the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the User Directory with the settings that follow:

Sample Type: Inorganic
Control Type: Ramp to Temperature
Power: 100%
Ramp: 5 minutes to 180 °C
Hold: 10 minutes

10.3.2 Fill each tube with 30mL of the nitric acid solution described in Section 7 and cap tightly. Place the tubes in the carousel, then run the "Clean Method"

10.3.3 After the cleaning method, allow the vessels to cool then dispose of the nitric acid in waste stream J. Rinse the vessel with DI water three times and then either allow the vessels to air dry or rinse with acetone to remove all water.

10.4 Assemble and Clean the Extraction Tubes Immediately Before Use.

10.4.1 If the microwave tube, cap, or plugs are wet, pre-rinse with acetone.

10.4.2 Rinse the microwave tube, cap and plug with methylene chloride. The plugs can be placed in a large Büchner funnel to help facilitate the rinse.

10.4.3 Discard the solvent in the correct waste stream.

10.5 Aliquot Samples

10.5.1 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. If the sample is a wipe, transfer the wipe to the extraction vessel.

10.5.2 Label microwave vessel with the sample ID, method, batch number, and date. The label needs to be flat and placed close to the bottom of the vessel.

10.5.3 Weigh 30 to 33 g of sample into the labeled microwave vessel.

10.5.4 For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled microwave vessels.

10.5.5 Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

NOTE: Care should be taken to ensure that the top lip of the tube is clean of any sample material or debris so that the plug will fit tightly later.

10.5.6 Place the microwave vessel on a cart next to the sample container so that

a second analyst can check the labels. This is documented on the Organic Extraction Checklists (See WI-DV-009).

10.6 Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

- Methylene Chloride is used for soil and wipe samples for the following methods:
  - SW-846 8015B (8015B_DRO)
  - SW-846 8015C (8015C_DRO)
  - Alaska Methods AK102 and AK103 (AK102_103)
  - NWTPH DRO (NWTPH_Dx)
  - Oklahoma DRO Method (Okla_DRO)
- For soil extraction by all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.
- For wipe samples by method 8081 and 8082, the solvent used is hexane.
- For wipe samples by method 8270 SIM, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.7 Add Surrogate and Spike Solutions

NOTE: The standards should be allowed to come to room temperature before spiking the samples.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

10.7.1 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples.

10.7.2 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard (see WI-DV-009) to the microwave vessel for each field sample and QC sample. Record the ID of the standard used on the benchsheet.

10.7.3 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard (see DV-OP-009) to the microwave vessel containing any LCS, LCSD, MS, and MSD samples. Record the ID of the standard used on the benchsheet.

10.8 Making sure not to overflow the vessel, slowly add approximately 30 mL of the appropriate solvent to the vessel. See Section 10.6 above for the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the surrogate and spiking standards to prevent loss of the more volatile compounds. For wipe samples add the solvent to the container that the wipe was received in and then transfer it to the microwave vessel. This is done to ensure a quantitative transfer of any solvent and material in the wipe sample container.

NOTE: The solvent should completely cover and saturate the sample so additional solvent may be needed depending on the matrix of the individual sample. The sample and solvent must not be higher than one inch below the threads of the vessel.

10.9 Seal the vessels by placing the plug on top of the vessel, small side down, and hand tighten the cap over the plug.

NOTE: Care should be taken to ensure that the plug, the cap, and the threads of the vessel are clean of any material or debris.

10.10 After sealed, the vessels must be inverted several times to ensure that the material is well mixed and saturated.

10.11 Load vessels into the carrousel.

10.11.1 There must be at least 8 vessels in the carrousel. Adding blank vessels with sand and solvent may be necessary.

10.11.2 Balance the tubes around the carrousel to ensure that all samples are exposed to an equal amount of energy during the extraction. For 8-16 samples, use only the inside ring. For 17-24 samples use only the outer ring. For more than 24 samples the inner ring should be filled completely first and additional samples balanced around the outer ring.

10.11.3 Only samples using the same extraction solvent should be placed in the same carrousel and ran at the same time.

10.11.4 For the vessels to be correctly loaded in the carrousel the cap should completely touch the top of the carrousel with nothing else of the extraction vessel visible.

10.12 Place the carrousel into the microwave, making sure that it sits on the turning apparatus correctly. The carrousel should be able to rotate. Close the door.

10.13 The Method Menu screen should indicate “Start Current Method” as being 3546 Full Xpress. Press the green “Start/Pause” button to begin the extraction.

NOTE: If a different method is shown, go to the “Load Method” on the menu screen. Choose “User directory” and place the cursor on the desired method. Press the “Home” button to return to the main menu, where the test highlighted will appear under the “Start Current Method”.

The method is under the User Directory with the settings that follow:

Sample Type: Organic
Control Type: Ramp to Temperature
Power: 100% (1600W)
Ramp: 20 minutes to 115 °C
Hold: 10 minutes

10.13.1 When the extraction is complete, the vessels will need to return to room temperature prior to opening the vessels. The microwave will indicate the

approximate temperature of the vessels.

CAUTION: Do not remove the carousel until the microwave is at room temperature.

10.13.2 The microwave contains a solvent sensor that will indicate the presence of solvent in the microwave and will pause the extraction. To minimize this, care needs to be taken not to overfill the vessel and to properly cap and tighten the vessel prior to extraction. If the solvent sensor indicates the presence of solvent, open the door and inspect the tops of the tubes for evidence of a solvent leak. Re-tighten the caps and start the extraction again.

10.14 Assemble and Clean Filter Funnels and Media Jars.

10.14.1 Without gloves on, fold a 15 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 250-mL media bottle.

10.14.2 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with the extraction solvent (see Section 10.6), so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.14.3 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.14.4 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.14.5 Discard the solvent in the correct waste stream.

10.15 Filter the Extracts

10.15.1 After the extraction method is complete and the vessels reach room temperature, quantitatively transfer the entire sample through solvent rinsed sodium sulfate funnels and into the media jar. The quantitative transfer is performed by rinsing the microwave extraction vessel at least three times with solvent.

10.15.2 Once the solvent has completely drained into the collection apparatus, rinse the funnel contents with 10 to 20 mL of additional solvent. Dispose of the solid sample and sodium sulfate into Waste Stream D and cap the media jar with the extract with a Teflon-lined lid or aluminum foil.

10.16 If the extract contains visible solids, it will be necessary to vacuum filter the extract prior to sending the extract on to concentration.

10.17 Store the extract refrigerated 4 ± 2 °C until concentration.

10.18 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

10.19 Clean the Teflon™ extraction vessels in hot soapy water (See DV-OP-0004 for how to prepare the soapy water). Use only a special sponge brush on the vessels to ensure they are not scratched. Rinse three times with hot tap water followed by two rinses of DI water. Then allow the vessels to air dry or rinse with acetone to remove all water.

10.20 All other glassware used in the procedure is washed according to DV-OP-0004.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

Before analyzing samples, the laboratory must establish a method detection limit (MDL). See DV-QA-005P, Determination of Method Detection Limits, for more information on the method detection limit studies.

13.2 Demonstration of Capabilities

An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See M-Q-001, TestAmerica Quality Management Plan, and the TestAmerica Denver Laboratory Quality Assurance Manual (QAM) for more information on the IDOCs.

13.3 Training Requirements

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 Flammable solvent – Waste Stream C

15.2.3 Solid waste/sodium sulfate – Waste Stream D

15.2.4 Nitric Acid Waste – Waste Stream J

15.2.5 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3456 Microwave Extraction, Revision 0, February 2007.

17.0 Method Modifications:

17.1 SW-846 Method 3546 calls for samples to be either air-dried and ground or mixed with sodium sulfate prior to extraction. This procedure does not call of the air-drying of samples unless requested by the client as this may lead to loss of the more volatile compounds. In addition, this procedure does not call for the use of sodium sulfate in the extraction vessel as the salt may cause the extract to overheat.

17.2 Method NWTPH-Dx calls for samples to be extracted by method SW-846 3550C. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method NWTPH-Dx is a possible determinative method by this procedure.

17.3 Method AK102 and AK103 calls for samples to be extracted by soxhlet. Valid MDLs and IDOCs have been completed using this procedure, therefore method AK102 and AK103 are listed as a possible determinative methods by this procedure.

17.4 Oklahoma Department of Environmental Quality DRO Method calls for samples to be extracted by sonication or soxhlet. Valid MDLs and IDOCS have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method Oklahoma Dept. of Environmental Quality DRO Method is a possible determinative method by this procedure.

17.5 Washington State Dept. of Ecology Method for the Determination of Extractable Petroleum Hydrocarbons Fractions calls for samples to be extracted SW-846 3550C, 3540C, or 3545. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable, therefore Method WA EPH is a possible determinative method by this procedure.

18.0 Attachments

Table 1: Determinative Methods Using Ultrasonic Extraction

19.0 Revision History

- Revision 1 dated 01 Jan 2011
  - Added 8270C SIM as a valid determinative method by microwave extraction.
  - Changed the procedure to call for the extract to be filtered thru a conical steel funnel lined with cellulose filter paper instead of a glass funnel with glass wool. This was done to help remove sediment from the extracts.
  - Removed details about the surrogate and spike standards used in the extraction. This information can now be found in DV-OP-0020.
  - Added instructions to Section 7 on how to prepare the nitric acid solution used in the weekly cleaning of the tubes.
  - Changed the solvent used in the extraction of samples for method 8081 and 8082. The samples are now extracted in a 1:1 Mixture of MeCl2:Acetone instead of a 1:1 Mixture of MeCl2:Hexane.
  - Revised the procedure in Section 10.5 for aliquotting samples to state that 30 to 33g of sample should be used instead of 30±2g and that the weight should be recorded to the nearest 0.1g instead of the nearest mg.
- Revision 0.1 dated 12 March 2010
  - Updated implementation date
  - Added section 6.1

TABLE 1.

Determinative Methods Using Ultrasonic Extraction

Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020 DV-GC-0026
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021 DV-GC-0030
Polynuclear Aromatic Hydrocarbons by HPLC	SW-846 8310	DV-LC-0009
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C NWTPH-Dx AK102 AK103 OK Dept. of Environ. Quality DRO Method	DV-GC-0002 DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002

Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Michael Flournoy 12/16/09 Technical Manager Date	Joe Schairer 12/16/09 Health & Safety Manager / Coordinator Date
Douglas Weir 12/18/09 Quality Assurance Manager Date	Karla Buechler 12/21/09 Laboratory Director Date

Facility Distribution No. _____ **Distributed To:_____**

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290 and 8290A. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques. Sample preparation is addressed in WS-IDP-0005.

2.2. One to two μL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.3. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective internal or recovery standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. All other identified PCDD/PCDF congeners are identified by their relative

retention times based on the daily CCV standard , and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.4. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.4. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.5. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.

3.6. Congener: Any isomer of any homologous series.

3.7. Internal Standard: An internal standard is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 3. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

3.8. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while ^{13}C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. ^{13}C-1,2,3,7,8,9-HxCDD also acts as a retention

time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.9. Estimated Detection Limit (EDL)/ Estimated Quantiation Limit (EQL): The sample specific estimated detection limit (EDL/EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.

3.10. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when 2,3,7,8-TCDF on the DB-5 column is detected.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material,

operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the 'Schedule of Routine Maintenance' in the QAM.

6.2. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.2.1. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2-μL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 μL). 1 μL injections are allowed; however, laboratories

are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.2.2. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.2.3. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.2.4. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.3. GC Column

6.3.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when 2,3,7,8-TCDF is detected.

6.3.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which

samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Distilled water demonstrated to be free of interferents

7.1.2. Potassium carbonate, anhydrous, analytical reagent.

7.1.3. Silica gel.

7.2. Solvents

7.2.1. High-purity, distilled-in-glass or highest available purity: methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, and acetone.

7.3. All calibration, daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.3.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.4. Calibration Solutions

7.4.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/µL) and the highest for the octachlorinated congeners (2000 pg/µL).

7.4.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard

solutions.

7.4.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

7.4.4. Standards for method 8290A require storage at ≤ 6°C.

7.5. GC Column Performance Check Solution

7.5.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.

7.5.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of ≤ 25%. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

7.6. Field Surrogate Solution (air matrices)

7.6.1. This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Methods 23 and/or 0023A) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.7. Sample Fortification Solution (Internal Standard)

7.7.1. This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C-OCDF is not present in the solution.)

7.8. Recovery Standard Solution

7.8.1. This tetradecane solution contains two recovery standards (^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,HxCDD). An appropriate volume of this solution will be

spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See SAC-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.7. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤ 6°C.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The

method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. The method blank must be spiked prior to extraction with the same amount of ^{13}C-labeled internal standards as added to samples.

9.1.2. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.1.2.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.2.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.2.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance

criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above upper control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. Analyze the MS and MSD samples as described in Section 11.

9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.7. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Surrogate/Clean Up Recovery Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.

9.6. Internal Standards

9.6.1. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.

9.6.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.6.3. A low or high percent recovery for a blank does not require discarding the

analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.7. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.

10.2. Tuning (Mass Resolution Check)

10.2.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.2.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

10.2.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). Verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

10.2.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760). The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 3) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3. Performance Checks

10.3.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 3 (HRCC-3) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-2 or HRCC-4 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.3.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must

demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.3.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C-HxCDF and ^{13}C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

10.3.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.4. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.4.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.4.2. Tune the instrument with PFK.

10.4.3. Inject 1 or 2 μL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.

10.4.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.4.3.2. The same injection volume must be used for all samples, QC, and standards.

10.4.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or 2-µL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

10.4.4.1. The total cycle time for data acquisition must be < 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

10.4.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.

10.4.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

10.4.4.4. The ratio of integrated ion current for the ions belonging to the ^{13}C labeled internal and recovery standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

10.4.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

10.4.5.1. Referring to Table 5, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate internal standards (Table 5) and the nine RRFs for the labeled ^{13}C internal standards [RRF(m); m=18 to 26] relative to the two recovery standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}} \qquad RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for the labeled internal standards,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled recovery standards,

Q_{is} = quantity of the internal standard injected (pg),

Q_{rs} = quantity of the recovery standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Qis, Qrs, and Qx must be the same.

10.4.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = (1/5)\sum_{j=1}^{5} RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 5), and j is the injection number (or calibration solution number; j = 1 to 5).

10.4.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

10.4.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain ^{13}C-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the [M+6]+ ion of ^{13}C-OCDF from the [M+2]+ ion of OCDD (and [M+4]+ from ^{13}C-OCDF with [M]+ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C-OCDD.

10.4.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = (1/t)\sum_{n=1}^{t} RRF_n$$

Where:

k = 27 to 30, with 27 = PeCDF;

28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.4.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RRF(m) = \frac{A_{is}^{m} \times Q_{rs}}{Q_{is}^{m} \times A_{rs}}$$

$$\overline{RRF}(m) = (1/5)\sum_{j=1}^{5} RRF_j(m)$$

Where:

m = 18 to 26 (congener type)

j = 1 to 5 (injection number),

A_{is}^{m} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),

Q_{rs} & Q_{is}^{m} = quantities of, respectively, the recovery standard (rs) and a particular internal standard (m) injected (pg),

RRF(m) = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and

____

RRF(m) = calculated mean relative response factor of a particular internal standard, as determined from the five initial calibration injections (j).

10.5. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

10.5.1. The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

Note: If Method 8290A criteria are required for the project then both the percent standard relative standard deviation for the mean response factors for the 17 unlabeled standards and the nine labeled reference compounds must be ≤ 20 percent.

10.5.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

10.5.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.6. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.6.1. Inject 1 or 2 μL of the concentration calibration solution HRCC-3 containing 10 pg/μL of tetrachlorinated congeners, 50 pg/μL of penta-, hexa-, and heptachlorinated congeners, 100 pg/μL of octachlorinated congeners, and the respective internal and recovery standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and

document an acceptable calibration as provided in Section 10.6.

10.7. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.7.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.7.1.1. The bracketing continuing calibration must be ± 20% of the average RRF calculated from the initial calibration.

10.7.1.1.1. If the target compounds in the ending standard are less than or equal to ± 20% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.7.1.1.2. If the target analytes are greater than ± 20% but less or equal to ±25% and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than ± 20% but less or equal to ±25% and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.7.1.1.3. If the percent deviation of unlabeled compounds exceeds ± 25%, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.7.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.7.2.1. The bracketing continuing calibration must be ± 30% of the average RRF calculated from the initial calibration.

10.7.2.1.1. If the labelled compounds in the ending standard are less than or equal to ±30% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.7.2.1.2. If the internal standard analytes are greater than ± 30% but less or equal to ±35%, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.7.2.1.3. If the percent deviation of labeled compounds exceeds ± 35%, reanalyze samples if adversely impacted.

10.7.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.7.4. If either criteria in Sections 10.6.1 or 10.6.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.3.4.4 for resolution.

10.7.5. If the above criteria (Section 10.6.1) cannot be satisfied, the entire initial calibration process (Section 10.3) must be repeated.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Dilution Procedure – Simple Dilutions

Dilutions from 2X to 100X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 50X dilution of original 10 g/20 µL sample

(10 g/20 µL) x (2 µL aliquot + 98 µL keeper) = 1 g/100 µL FV

Record the final sample concentration on the extract label.

11.3. Sample Dilution Procedure – Complex Dilutions

Complex dilution requiring respiking of IS and RS: Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL RS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

11.4. Analytical Procedures

11.4.1. Inject a 1 or 2 µL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.

11.4.2. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

12. CALCULATIONS/DATA REDUCTION

12.1. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.1.1. Retention Times

12.1.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled internal or recovery standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.1.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract, the relative

retention time (relative to the appropriate internal standard) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C-OCDD as determined from the daily routine calibration results.

12.1.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.1.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.1.1.5. The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.1.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

12.1.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 4 describes the procedure to be followed for the determination of the S/N.

12.1.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12.2. For gas chromatographic peaks that have met the criteria outlined above, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times RRF(n)}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

Ax = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

Ais = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

Qis = quantity, in pg, of the internal standard added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).

RRF(n) = Calculated mean relative response factor for the analyte [RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

12.3. Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Percent Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times RRF(m)} \times 100$$

Where:

Ais = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

Ars = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

Qis = Quantity, in pg, of the internal standard added to the sample before extraction,

Qrs = Quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

RRF(m) = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

12.4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

12.4.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified

appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.4.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.5. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.

12.5.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.

12.5.2. Extraction of an aliquot large enough to be representative with an increased concentration of internal standard and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.

12.5.3. Dilution of the original extract. Internal standard components are re-spiked at an appropriate level prior to analysis. In this case, the internal standard recoveries are taken from the original analysis.

12.6. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.

12.7. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

12.8. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) or estimated quantiation limit (EQL, 8290A) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL/EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.8.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression for EDL/EQL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL/EQL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their internal standard must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific\,2,3,7,8-subst.PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs. (also EQL for Method 8290A)

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{is} = height of one of the quantitation ions (Table 6) for the labeled internal standards.

W, RRF (n), and Qis retain the same meanings as defined in Section 12.2

12.8.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 11.8.4, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDLEQL can be calculated using the above formula and the height of one of the ions as appropriate.

12.9. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.10. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 10). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

12.11. Two-GC Column TEF Determination

12.11.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq$ 25% valley.

12.11.2. For samples that have a positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result above the lower calibration limit is calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be < 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.11.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.11.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 11 and the results from the routine

calibration run on the DB-5 column.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.5. GC Column Performance

13.5.1. Inject 1 or 2 μL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of < 1 second.

13.5.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of $\leq$ 25

percent (Figure 2),

Where:

$$\text{Valley Percent} = (x/y) \times 100$$

x = measured as in Figure 2 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

13.5.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

13.5.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials containing assorted solvents and extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Modifications from EPA 8290 and EPA 8290A

17.1.1. The methods specify that 2 µL injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 µL injections for all performance checks, standards, QC samples, and samples.

17.1.2. In Section 2.7 of Method 8290 and 8290A, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled internal standards. All available labeled internal standards are used; therefore, a retention time window of -1 to +3

seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290 and 7.9 of Method 8290A.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.2. Modifications from TO-9A method

17.2.1. The ^{37}CL-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.2. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.2.3. The final volume is adjusted to 20 µL in tetradecane.

17.2.4. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of the Sample Fortification and Recovery Standard Solutions.

18.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

18.5. Table 5 - Concentrations of Calibration Solutions

18.6. Table 6 - Ions Monitored for PCDDs/PCDFs

18.7. Table 7 - Recommended GC Operating Conditions

18.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

18.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

18.10. Table 10 - 2,3,7,8-TCDD Equivalent Factors

18.11. Table 11 - TEF: Analyte Relative Retention Time Reference Attributes

18.12. Figure 1 - Compound Structure

18.13. Figure 2 - GC Performance Check Chromatogram on the DB-5 Column

18.14. Figure 3 - PFK Peak Profile

18.15. Figure 4 - Manual Determination of Signal-to-Noise

18.16. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-ID-0005, Revision 7.3, Effective 12/30/2009

19.1.1. Editorial revisions.

19.2. WS-ID-0005, Revision 7.2, Effective 11/02/2009

19.2.1. Section 6.1: Inserted “Preventive and routine maintenance is described in the ‘Schedule of Routine Maintenance’ in the QAM.”

19.2.2. Section 12.1.2: Removed the word "presumptive" and inserted "above the lower calibration limit" after the word result.

19.3. WS-ID-0005, Revision 7.1, Effective 09/04/2009

19.3.1. Added Section 1.5, “When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.”

19.3.2. Inserted Section 10.1, “For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 “Calibration Curves (General)”.”

19.4. WS-ID-0005, Revision 7, Effective 10/02/2008

19.4.1. Updated to TestAmerica format.

19.4.2. Updated Table 9.

19.4.3. Sample preparation moved to SOP WS-IDP-0005

19.4.4. Added 8290A references.

19.4.4.1. Estimated Quantitation Limit (EQL).

19.4.4.2. Extract and standard storage.

19.4.4.3. Removal of MS/MSD.

19.4.4.4. Change to calibration criteria for labeled compounds.

19.4.5. Editorial changes.

19.5. WS-ID-0005, Revision 6.7, Effective 8/21/2008

19.5.1. Changed the word “toluene” to “acetone” in 7.11.2.

19.6. WS-ID-0005, Revision 6.6, Effective 4/9/2008

19.6.1. Added South Carolina rule to prepare an MS/MSD with every batch.

19.6.2. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

19.7. SAC-ID-0005, Revision 6.5, Effective 1/09/2007

19.8. SAC-ID-0005, Revision 6.4, Effective 08/29/2005

19.9. SAC-ID-0005, Revision 6.3, Effective 9/20/2004

19.10. SAC-ID-0005, Revision 6.2, Effective 02/24/2004

19.11. SAC-ID-0005, Revision 6.1, Effective 11/19/2003

19.12. SAC-ID-0005, Revision 6, Effective 12/15/2002

19.13. SAC-ID-0005, Revision 5, Effective 7/13/2001

19.14. SAC-ID-0005, Revision 4, Effective 9/11/1998

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (μL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Recovery Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/µL; Solvent: Isooctane	Recovery Standard Solution Concentration pg/µL; Solvent: Tetradecane
^{13}C-2,3,7,8-TCDD	2[a], 100[c]	--
^{13}C -2,3,7,8-TCDF	2[a], 100[c]	--
^{13}C -1,2,3,4-TCDD	--	100
^{13}C -1,2,3,7,8-PeCDD	2[a], 100[c]	--
^{13}C -1,2,3,7,8-PeCDF	2[a], 100[c]	--
^{13}C -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
^{13}C -1,2,3,7,8,9-HxCDD	--	100
^{37}Cl-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
^{13}C -2,3,4,7,8-PeCDF[c]	100[c]	
^{13}C -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
^{13}C -1,2,3,4,7,8-HxCDD[c]	100[c]	
^{13}C -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
^{13}C -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
^{13}C -OCDD	4[a], 200[c]	--

(a) Standard 8290, 8290A, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) ^{13}C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and ^{13}C -1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an internal standard.
(+)The ^{13}C -labeled analog is used as a recovery standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

TABLE 5

High Resolution Concentration Calibration Solutions

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS1**	**CS2**	**CS3 (VER(6))**	**CS4**	**CS5**
	Native CDDs and CDFs					
1	2,3,7,8-TCDD	0.5	2	10	40	200
2	2,3,7,8-TCDF	0.5	2	10	40	200
3	1,2,3,7,8-PeCDD	2.5	10	50	200	1000
4	1,2,3,7,8-PeCDF	2.5	10	50	200	1000
5	2,3,4,7,8-PeCDF	2.5	10	50	200	1000
6	1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
7	1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
8	1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
9	1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
10	1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
11	1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
12	2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
13	1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
14	1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
15	1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
16	OCDD	5.0	20	100	400	2000
17	OCDF	5.0	20	100	400	2000
	Labeled CDDs and CDFs					
18	$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
19	$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8,9-	100	100	100	100	100

	Compound	**Concentration (ng/mL)**				
RRF (n)(m)		**CS1**	**CS2**	**CS3 (VER(6))**	**CS4**	**CS5**
	HpCDF					
26	$^{13}C_{12}$-OCDD	200	200	200	200	200
	Cleanup Standard/ FS					
	$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
	Recovery Standards					
	$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 6*

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate[(a)] Mass	Ion ID	Elemental Composition	Analyte
1	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9338	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	[330.9792]	LOCK	C_7F_{13}	PFK
2	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
	[342.9792]	LOCK	C_8F_{13}	PFK
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8156	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)
	[380.9760]	LOCK	C_8F_{15}	PFK

TABLE 6 (cont.)*

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate[(a)] Mass	Ion ID	Elemental Composition	Analyte
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7788	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8250	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	423.7767	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}CL_7{}^{37}Cl_2O$	NCDPE
	[430.9728]	LOCK	C_9F_{17}	PFK
5	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7780	M+2	$^{13}C1_2{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	471.7750	M+4	$^{13}C1_2{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)
	513.6775	M+4	$^{13}C1_2{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE
	[442.9728]	LOCK	$C_{10}F_{17}$	PFK

[(a)] The following nuclidic masses were used:

H =	1.007825	O =	15.994915
C =	12.000000	^{35}Cl =	34.968853
^{13}C =	13.003355	^{37}Cl =	36.965903
F =	18.9984		

S = Internal/recovery standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C
Interface Temperature: 280°C
Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column[(b)]

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[(a)]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

[(a)] In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- $^{13}C_{12}$-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are $\leq$ 25%.

TABLE 9

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[(a)]	M / M+2	0.51	0.43	0.59
7[(b)]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

[(a)] Used only for ^{13}C-HxCDF (IS) [(b)] Used only for ^{13}C-HpCDF (IS)

TABLE 10

2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

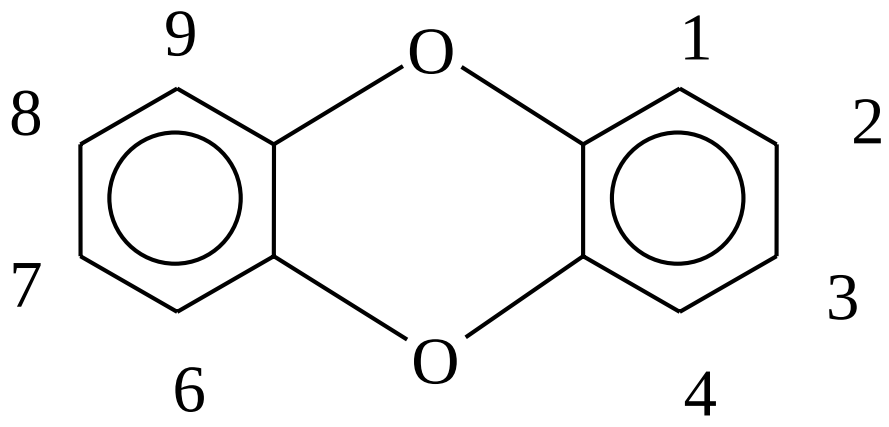
TABLE 11

Toxicity Equivalency Factor:
Analyte Relative Retention Time Reference Attributes

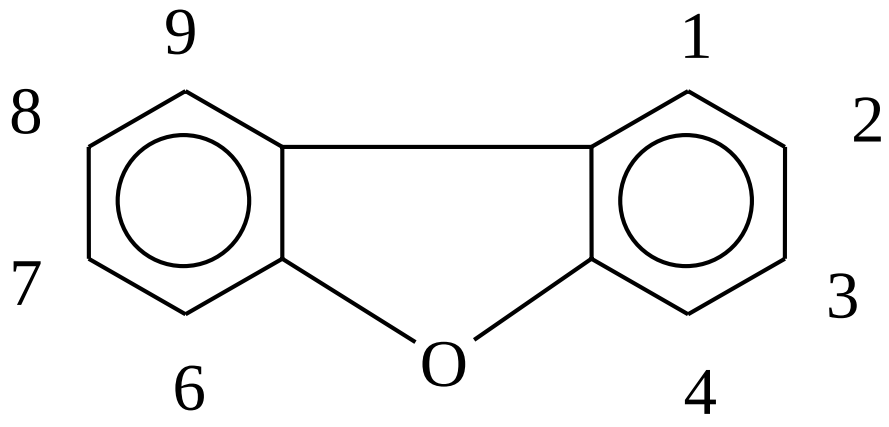
Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF

FIGURE 1
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

FIGURE 2

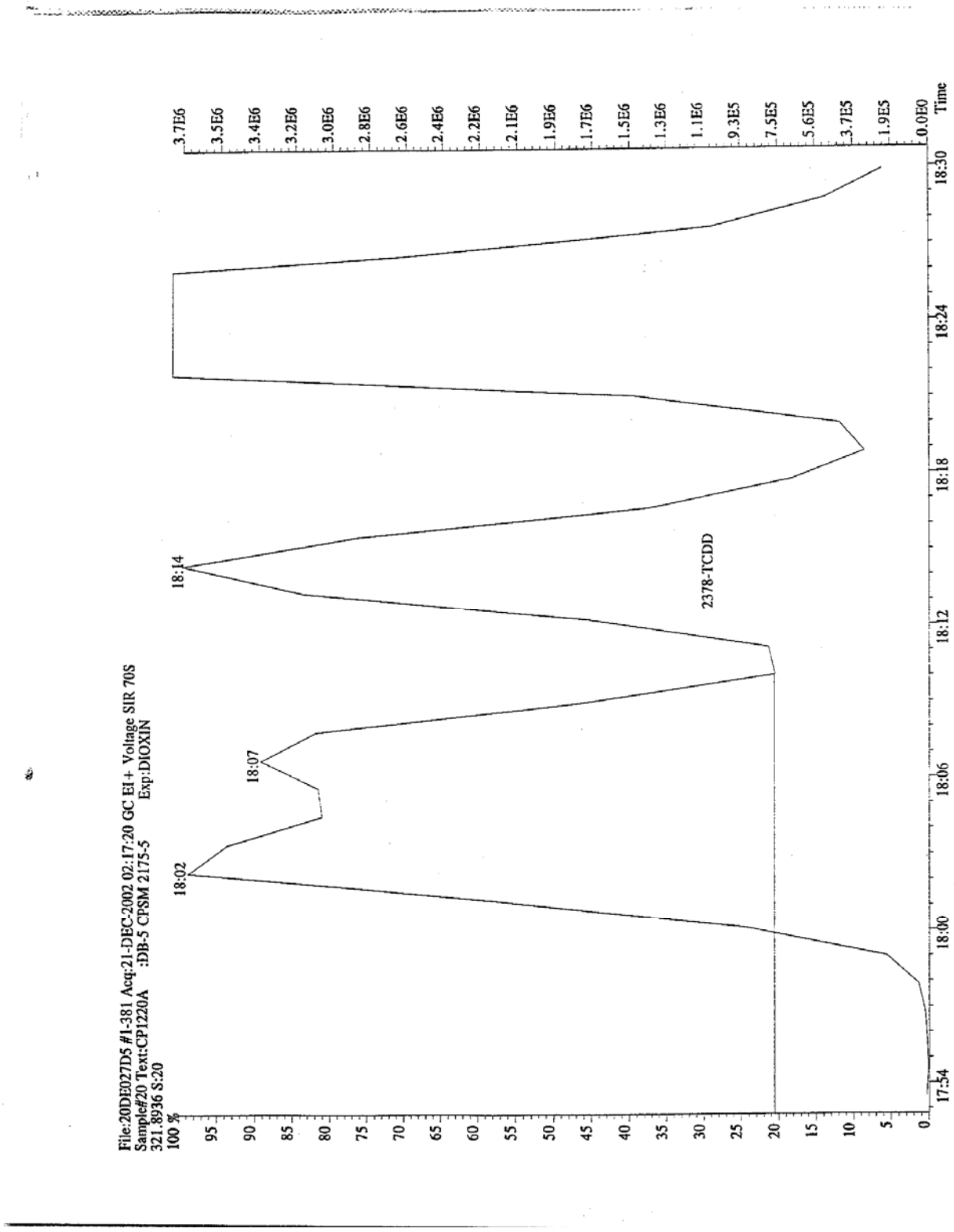

File:20DE027D5 #1-381 Acq:21-DEC-2002 02:17:20 GC EI+ Voltage SIR 70S
Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time
17:54
18:00
18:06
18:12
18:18
18:24
18:30
3.7E6
3.5E6
3.4E6
3.2E6
3.0E6
2.8E6
2.6E6
2.4E6
2.2E6
2.1E6
1.9E6
1.7E6
1.5E6
1.3E6
1.1E6
9.3E5
7.5E5
5.6E5
3.7E5
1.9E5
0.0E0

Figure 3

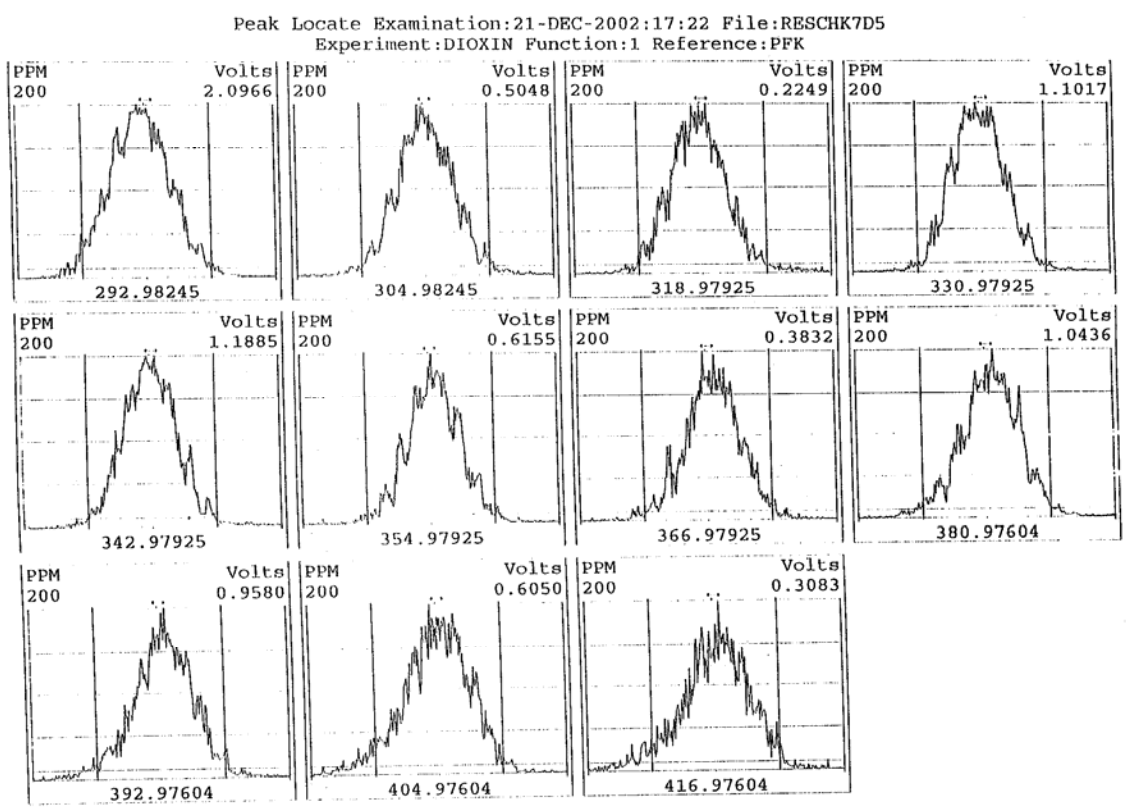
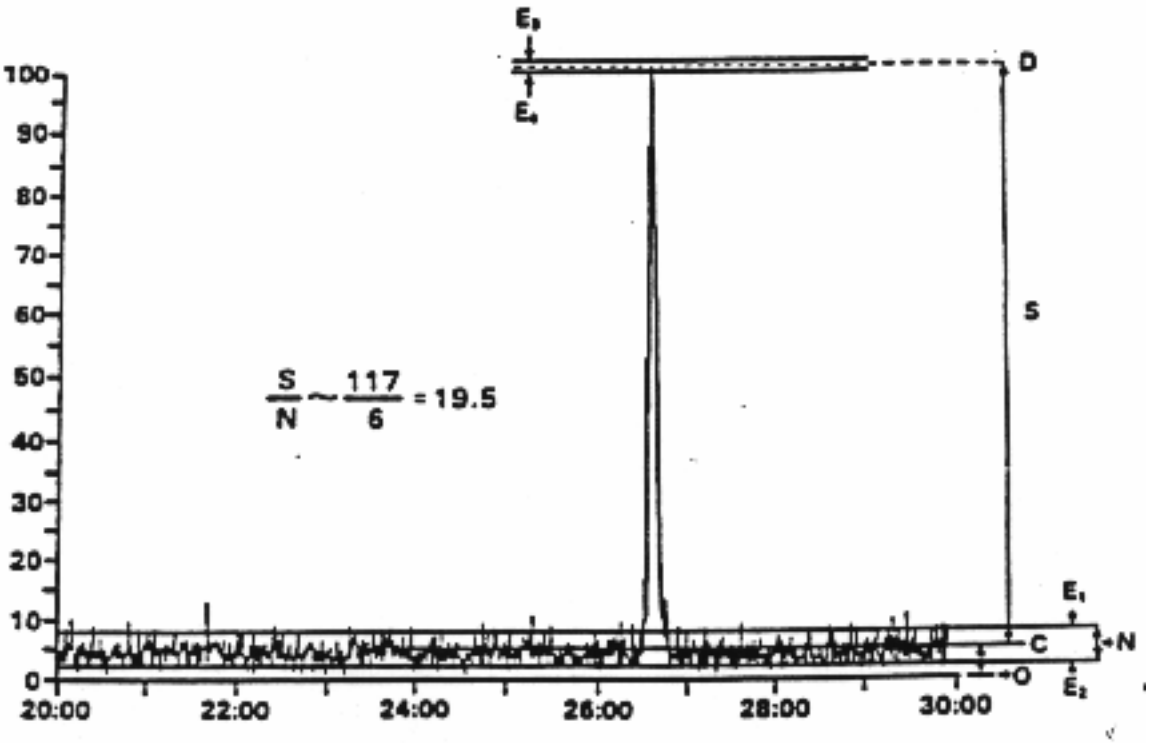

Peak Locate Examination:21-DEC-2002:17:22 File:RESCHK7D5
Experiment:DIOXIN Function:1 Reference:PFK
PPM 200 Volts 2.0966
292.98245
PPM 200 Volts 0.5048
304.98245
PPM 200 Volts 0.2249
318.97925
PPM 200 Volts 1.1017
330.97925
PPM 200 Volts 1.1885
342.97925
PPM 200 Volts 0.6155
354.97925
PPM 200 Volts 0.3832
366.97925
PPM 200 Volts 1.0436
380.97604
PPM 200 Volts 0.9580
392.97604
PPM 200 Volts 0.6050
404.97604
PPM 200 Volts 0.3083
416.97604

FIGURE 4



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

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November 1992

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 μL (either in a minivial or in a capillary tube). Inject 2 μL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

Title: Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Steve Rogers 1/14/10 Date Technical Manager	Joe Schairer 1/18/10 Date Health & Safety Manager / Coordinator
Douglas Weir 01/19/10 Date Quality Assurance Manager	Karla Buechler 1/19/2010 Date Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the preparation of samples prior to the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Refer to Table 1 for the list of analytes. Analysis is by SOP WS-ID-0005.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis.

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction and analyte-specific cleanup techniques.

2.2. A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent) extraction, for soil, sediment, fly ash samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction for water samples; c) dilution of a small sample aliquot in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent) extraction for fish tissue.

2.3. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20 µL of a tetradecane solution containing 100 pg/µL of each of the two recovery standards $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF internal standards while the latter is used for the determination of hexa-, hepta- and octa-

chlorinated PCDD/PCDF internal standard percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Internal Standard: An internal standard is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 2. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

3.4. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.5. Cleanup Recovery Standard (CRS): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are chlorinated-biphenyls, methoxy biphenyls, hydroxy biphenyl ethers, benzyl phenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in Sections 11.11 thru 11.15 can be used to reduce or eliminate these interferences.

4.4.1. If South Carolina samples show diphenyl ethers at levels that could contribute to positive furan hits, a subsequent clean-up to remove them must be performed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Hearing protection must be worn when using mechanical systems to grind fish, tissue, or paper/pulp samples.

5.1.2. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.3. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.7. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. The use of separatory funnels during the partition and back extraction of sample extracts can also create excessive pressure. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the extraction can be performed behind a closed fume hood sash on a mechanical shaker.

5.1.8. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Isooctane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Potassium Hydroxide	Corrosive Poison	2 mg/m3 ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on the severity of exposure. Symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.1. Nitrogen evaporation apparatus with variable flow rate.

6.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3. Centrifuge.

6.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2ºC.

6.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.6. Drying oven.

6.7. Stainless steel spoons and spatulas.

6.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.

6.10. Reacti-vial, 2 mL, silanized clear glass.

6.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.12. Separatory funnels, 250 mL.

6.13. Separatory funnels, 1000 mL.

6.14. Teflon® boiling chips (or equivalent) washed with methlyene chloride before use.

6.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.16. Adapters for concentrator tubes.

6.17. Glass fiber filters.

6.18. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.19. Continuous liquid-liquid extractor.

6.20. All-glass Soxhlet apparatus, 500 mL flask.

6.21. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.

6.22. Glass funnels, sized to hold 170 mL of liquid.

6.23. Desiccator.

6.24. Turbo evaporator

6.25. Rotary evaporator with a temperature controlled water bath.

6.26. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.27. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:

6.28. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.

6.28.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

6.28.2. After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

6.28.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

6.28.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

7. REAGENTS AND STANDARDS

7.1. Column Chromatography Reagents

7.1.1. Silica Gel - Kieselgel 60 or equivalent, activate for 1 hour at 184°C before use. Store at 130°C in covered flask.

7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.

7.1.3. Basic Alumina - ICN or equivalent. No activation required.

7.1.4. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon ® lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.1.5. 44% H_2SO_4 /silica gel - Mix 24 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.1.6. 33% NaOH/silica gel - Mix 34 mL 1N NaOH and 67 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2. Acid Alumina Activity Assessment

Alumina activity may vary with the matrix or environmental conditions. Monitor internal standard and cleanup recovery standard recoveries in extract analysis. Low recoveries of cleanup recovery standard (CRS) may indicate loss of alumina activity. Assess stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile).

Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified.

7.2.1. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:

7.2.1.1. Set up and label 3 acid alumina columns.

7.2.1.2. Pre-rinse with 20 mL hexane.

7.2.1.3. Add 2 mL hexane spiked with internal standards and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.

7.2.1.4. Elute each column with 20 mL hexane. Collect and label these fractions.

7.2.1.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.

7.2.1.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.

7.2.2. Review data and select an elution scheme. Group the fraction from each solvent system as follows:

7.2.2.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.

7.2.2.2. Analyte fraction - consists of all that contain detectable levels of target analytes.

7.2.2.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.

7.2.3. Select the solvent system which best meets the following two conditions:

7.2.3.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.

7.2.3.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

7.2.4. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

7.2.5. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

7.3. Reagents

7.3.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.3.2. Potassium hydroxide, ACS grade, 20 percent (w/v) in distilled water.

7.3.3. Distilled water demonstrated to be free of interferents

7.3.4. Potassium carbonate, anhydrous, analytical reagent.

7.3.5. Silica gel.

7.3.6. Solution for breaking emulsions: Slowly add 1.0L of reagent grade NaOH solution to a 2.0L NaOH container, containing 1.0L of DI H2O, and leave the container in secondary containment with the lid off.

Warning: The solution will begin to heat so let the solution stand until equilibrium is met and the solution is at room temperature.

When this process is complete, the solution will then be ready for use in the samples.

7.3.7. Precleaned Sodium Sulfate.

7.3.8. Canola Oil (for tissue extraction only), or other suitable oil.

7.4. Desiccating Agent

7.4.1. Sodium sulfate, granular, anhydrous.

7.5. Solvents

7.5.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

7.6. All daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.6.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.6.2. Standards for method 8290A require storage at $\leq 6^{o}C$.

7.7. Field Surrogate Solution (air matrices)

This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.8. Internal Standard

This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}C_{12}$ -OCDF is not present in the solution.)

7.9. Native Spike Standard

Also known as the Matrix Spike or Native Spike solution. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 2. Prepare using the appropriate standards to yield a spiking solution with a concentration of 4.0 ng/ml for the tetra-CDDs/CDFs, 20 ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40 ng/ml for the octa- CDD/CDF.

7.10. Recovery Standard Solution

This tetradecane solution contains two recovery standards ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$ -1,2,3,7,8,HxCDD). An appropriate volume of this solution is spiked into each sample extract before the final concentration step.

7.11. Cleanup Recovery Standard Solution (CRS)

Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 2, in isooctane (or toluene).

7.12. Preparation and QC of PUF material

7.12.1. The PUF material is purchased pre-cut.

7.12.2. The PUFs are rinsed by Soxhlet with acetone (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.

7.12.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.

7.12.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.

7.12.5. The 1613/8290 daily internal standard solution is spiked into the PUF and it is extracted for a minimum of 16 hours.

7.12.6. The Soxhlet extract is recovered and processed according to Section 11.4.

7.12.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. Grinding or blending of fish samples.

If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded.

Warning: Hearing protection must be worn when grinding samples.

8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤6ºC.

8.9. For moisture determinations refer to SOP WS-OP-0013.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory

matrix (reagent water, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.1.2. Use sodium sulfate as the method laboratory matrix when solids are extracted. Use a mixture of sodium sulfate and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.1.3. The method blank must be spiked prior to extraction with the same amount of ^{13}C -labeled internal standards as added to samples.

9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.1.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration,

then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air

samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.6. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1 L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of internal standard to yield 100 pg/μL in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11. As applicable, add the appropriate amount of recovery standard to yield 100 pg/μL in the final extract. Analyze a 1-2 μL aliquot of the concentrated extract using SOP WS-ID-0005.

9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of internal standard to yield 100 pg/μL in the final extract.

9.6.3. Using appropriate methods, concentrate to approximately 10 mL.

9.6.4. Just before analysis, add the appropriate amount of recovery standard to yield 100 pg/μL in the final extract. Reduce the volume to a final volume of 20 μL, as necessary. No column chromatography is required.

9.6.5. Analyze an aliquot following the same procedures used to analyze samples.

9.7. Surrogate/Clean Up Recovery Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.

9.8. Internal Standards

An internal standard is a ^{13}C -labeled analog of a PCDD/PCDF congener. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and

furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

9.8.1. A 2000 pg aliquot of the internal standard mixture is added to all samples, regardless of sample size. As an example, for $^{13}C_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of $^{13}C_{12}$ -2,3,7,8-TCDD to give the requisite fortification level.

9.8.2. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.

9.8.3. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.8.4. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.9. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$ -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

9.10. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample aliquots.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

10.1. On a daily basis, calibrate any balance to be used in accordance with SOP WS-QA-0041.

10.2. On a monthly basis, calibrate any autopipettor to be used in accordance with SOP WS-QA-0004.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Refer to SOP WS-ID-0009 for the preparation of stationary source samples.

11.3. Sample Pre-Treatment

11.3.1. Paper Pulp Sludges are generally air-dried and ground prior to extraction following Section 11.5. Because of the drying procedure, a Dean-Stark water separator is optional for extraction.

11.3.2. Fly Ash — Fly ash samples are pretreated with HCl prior to extraction by both soxhlet and separatory funnel techniques.

11.3.2.1. Weigh 2-10g of sample aliquot into a clean glass jar.

11.3.2.2. Add 1.0mL of the internal standard mixture with 2 mL of acetone.

11.3.2.3. Add 150 mL of 1N hydrochloric acid and shake for 4 hours.

11.3.2.4. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking.

11.3.2.5. Filter the contents of the jar through a glass fiber filter.

11.3.2.6. Extract the solids as per Section 11.5, omitting the daily internal standard spike for the samples.

11.3.2.7. Extract the aqueous filtrate as per Section 11.8, using 100 mL of toluene for the first shake, and 100 mL of hexane for subsequent shakes.

11.3.2.8. Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.11 as necessary.

Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4. Waste Dilution (Still-Bottom/Fuel Oil, and other solvent-miscible materials).

11.4.1. Weigh 1 g of the waste (organic liquids, fuel oils, and solids that will dissolve in a solvent) into a vial.

11.4.2. Add 40 mL of toluene (or other solvent if the material is not miscible/soluble in toluene). Shake gently to dissolve.

11.4.3. Remove a 4.0 mL aliquot (0.1g sample equivalent) and place in a culture tube. Add 1.0 mL of daily internal standard and 1.0 mL of cleanup recovery standard, and proceed to Section 11.11.

11.5. Soxhlet Extraction (Solids, Tissues, Sludges, Wipes)

11.5.1. Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene per second will fall from the condenser tip into the receiver. Extract the apparatus for a minimum of four hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.2. After pre-extraction, cool and disassemble the apparatus.

11.5.3. If tissues requiring % Lipids are to be extracted, for each sample weigh the concentration vessel with label and boiling chips. Record the mass on the benchsheet.

11.5.4. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.5.4.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.5.5. Place the thimble a Soxhlet apparatus equipped with a Dean-Stark water separator.

11.5.6. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.5.7. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.5.8. Reassemble the pre-extracted apparatus and add a fresh charge (250-300 mL) of toluene to the receiver and reflux flask.

11.5.9. Reflux 16 hours, with the solvent cycling at least 5 times per hour.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.10. Drain the water from the receiver if the receiver fills with water. Check and drain when necessary.

Note: If the receiver holds 10 mL of liquid, and 20 g of an approximately 10% solid sample is being extracted, then approximately 9 mL of water will end up in the receiver. In this case, the receiver will not need to be emptied (insufficient liquid to overflow), but it should be checked. If the sample amount is 50, and the percent solids is still 10%, then 45 mL of water will end up in the receiver. In this case, frequent checking is required, and the receiver will need to be emptied at least 5 times.

11.5.11. After refluxing, allow the apparatus to cool.

11.5.12. If samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.5.13. Proceed to Section 11.16.

11.6. SoxTherm Extraction (Solids, Tissues, Sludges,Wipes)

11.6.1. Prior to loading samples, run the system through a cleaning cycle (approximately 1 hour).

11.6.2. After pre-extraction, cool and disassemble the apparatus.

11.6.3. If tissues requiring % Lipids are to be extracted, for each sample weigh the vessel with label and boiling chips. Record the mass on the benchsheet. (If using the Soxtherm, weigh the 35 mm culture tube).

11.6.4. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise

specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.6.4.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.6.5. Place the thimble into the Soxtherm apparatus.

11.6.6. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.6.7. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.6.8. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus.

11.6.9. Program the system to boil for 1 hour, and reduce the toluene volume by 70-90 mL (volume < volume of the thimble).

11.6.10. Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL.

11.6.11. After refluxing, allow the apparatus to cool.

11.6.12. Pour the samples into round bottom flasks, and if samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.6.13. Proceed to Section 11.16.

11.7. Extract Splitting (Wipes)

Wipe extracts prepared using either Soxhlet or shaking techniques are split prior to further workup, to permit an archive aliquot, or analysis by an additional method. Once the extract has been concentrated using the rotovap or Turbovap, proceed as follows:

11.7.1. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or

appropriate volume) with the addition of rinse solvent.

11.7.2. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount as instructed by the client, program manager or department manager) and transfer to a culture tube. Archive the remaining sample for future use.

11.7.2.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.7.2.2. If "N" analyses are required, then the extract is divided into "N+1" equal portions, so that one portion is archived, and a portion is used for each test.

11.8. Aqueous Samples.

11.8.1. Weigh the sample in the bottle on the top loading balance to +1g, and record the mass. Mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Pour the entire sample (approximately 1 L) into a 2 L separatory funnel.

11.8.2. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.8.3. Create a blank and LCS by adding 1 L of laboratory reagent water to 2 additional separatory funnels. Add 100 mL methylene choride to each funnel.

11.8.4. For each sample, add 1 mL of daily internal standard solution into 2 mL of acetone. Add this solution to the sample in the separatory funnel. Each aliquot of spike mixture is added similarly.

11.8.5. To the LCS, add 50 µL of the precision and recovery standard dissolved into 2 mL of acetone.

11.8.6. Extract the samples by shaking each funnel for two minutes with periodic venting.

Warning: Separatory funnel extraction with methlyene chloride is a high-risk activity. Pressure may build rapidly in the funnel. It should be vented after several seconds of shaking, and often enough to prevent build-up of pressure. Chemist performing separatory funnel extraction must wear a face shield over their safety glasses/goggles. Alternatively, the extraction can be performed behind a closed fume hood sash.

11.8.7. Allow the organic layer to separate from the water phase for a minimum of

10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

11.8.8. Repeat the extraction two additional times with methylene chloride.

11.8.9. Determine the original sample volume by re-weighing the sample bottle, or filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11.8.10. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with methlyene chloride and load funnel with methlyene chloride -rinsed Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO with fresh methlyene chloride and collect in round bottom flask.

11.8.11. Transfer the extract to a 500-mL round-bottom, add approximately 100 µL of tetradecane and concentrate on a rotary evaporator or TurboVap.

11.8.12. Perform macro concentration as detailed in Section 11.16.

11.9. Breaking Emulsions

There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with methlyene chloride. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 1:1 $NaOH/H_2O$ solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. See Section 7.3.6 for reagent preparation.

11.9.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.

11.9.2. Pour approximately 100 mL of the 1:1 $NaOH/H_2O$ into a 1 L amber glass bottle (AGB).

11.9.3. Drain the sample with the emulsion from the 2 L separatory funnel into the 1 L AGB and let it stand.

11.9.4. Empty the aqueous waste into the LLE waste drum.

11.9.5. Pour the solution with methlyene chloride back into the same 2 L separatory funnel and drain the methlyene chloride phase through Na_2SO_4 into a 500 mL round-bottom flask.

11.9.6. Empty the aqueous waste into the LLE waste drum.

11.9.7. Proceed with macro concentration (Section 11.16).

11.10. Filter/PUF Samples

11.10.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.10.2. Add 2 mL (4000 pg) of 1613/8290 daily Internal Standard solution to all samples and QC.

11.10.3. Add 50 uL of 1613/8290 Native Spike to the LCS.

11.10.4. Extract the samples and QC for a minimum of 16 hours.

11.10.5. Concentrate the extract from the round bottom flask with hexane and adjust the volume.

11.10.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.10.7. Split the extract 50:50 for analysis and archive.

11.10.8. Proceed to Section 11.11.

11.11. Extract Clean-Up

11.11.1. For all samples which are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC Extracts (See also Section 9.71)

11.11.2. Proceed with further cleanups as dictated by the sample matrix and extract color. The “Option C” cleanup (Section 11.12) and the IFB Upper Column cleanup (Section 11.13) are applied to samples with high levels of interferences. The IFB column cleanup (Section 11.14) is applied to all samples.

11.12. Acid Partitioning (“Option C”)

11.12.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.12.2. Partition the extract in 50-125 mL of hexane against 40 mL concentrated H_2SO_4 in a separatory funnel. Shake for two minutes. Remove and discard

the H_2SO_4 layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

11.12.3. Partition the extract against 50 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15 mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.) The DI H_20 partition is applied only as samples warrant it at the discretion of the analyst.

11.13. IFB Upper Column Cleanup

11.13.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.13.2. Set up the upper of the two chromatography columns as depicted in Figure 2. The column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate.

11.13.3. Pre-rinse the column with 20 mL hexane, and discard the rinsate.

11.13.4. Add extract to the column. Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.13.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.13.6. Collect the eluate, and concentrate before proceeding with the IFB cleanup (Section 11.14).

11.14. IFB Column Cleanup

Most samples will undergo this cleanup, either direction following concentration on the rotovap, or following the cleanup in Section 11.12 (Option C) or Section 11.13 (IFB Upper Column).

11.14.1. Set up two chromatography columns as depicted in Figure 2. The upper column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g

sodium sulfate. The lower column (15 mm diameter) is packed in this order: a glass wool plug, 6 g acid alumina, and 1 g sodium sulfate.

11.14.2. Pre-rinse each column with 20 mL hexane, and discard the rinsate.

11.14.3. Put one column above the other.

11.14.4. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.14.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.14.6. Discard upper column.

11.14.7. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.14.8. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.14.9. Proceed with additional cleanups as necessary.

11.15. Carbon Column Clean-up (D2 Column)

Prepare an activated Carbon & Silica Gel column as described in below. Refer to the diagram in Figure 3 as well.

11.15.1. Push a glasswool plug down to the 3 inch mark in a pre-cut D2 column.

11.15.2. Add 1 g of 5% activated carbon/silica. Top with a glasswool plug.

11.15.3. With the column oriented with “A” on the top (and the carbon on the lower end of the column), pre-elute with 5 mL 1:1 methlyene chloride :cyclohexane.

11.15.4. Turn over (so that the “B” end is on top, and the carbon is now on the upper end of the column) and pre-elute with 5 mL 1:1 methlyene chloride :cyclohexane.

11.15.5. Discard pre-eluates.

11.15.6. Dilute the extract to 1 mL with hexane and transfer to the column (still oriented in the “B” direction).

11.15.7. Rinse sample vial onto the column with 2 x 2 mL 1:1 methlyene chloride:cyclohexane.

11.15.8. Elute with 6 mL 1:1 methlyene chloride :cyclohexane

11.15.9. Elute with 5 mL 75:25 methlyene chloride:methanol

11.15.10. Discard eluates.

11.15.11. Turn the column over (so that the "A" end is on top), and elute with 30 mL of toluene. Collect this eluate.

11.15.12. Concentrate to NEAR dryness using the Rotovap (Section 11.16) or Turbovap (Section 11.17), then proceed to the recovery standard step (Section 11.18).

11.16. Macro-concentration (Rotary Evaporator)

Concentrate the extracts in separate round bottom flasks on rotary evaporator.

11.16.1. Assemble the rotary evaporator according to manufacture's instructions, and warm the water bath. On a daily basis, preclean the rotary evaporator by solvent rinsing. Between samples, 2-3 rinses of toluene followed by a 2-3 mL rinse of hexane should be rinsed down the feed tube into a waste beaker.

Rotovap Conditions		
Solvent	**Bath Temperature (C)**	**Vacuum Setting (PSI)**
Toluene	80	25
Hexane	65	15
Methylene Chloride	70	No vacuum applied

11.16.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

11.16.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

11.16.4. For samples requiring % Lipids analysis:

11.16.4.1. Concentrate until the toluene has been completely removed. Add approximately 25 mL hexane and concentrate to ensure that only the lipids are present.

11.16.4.2. Dry the concentration vessel and let stand at room temperature. Weigh the vessel and record on the benchsheet.

11.16.4.3. Calculate % lipids as follows:

$$\% \text{ Lipids} = \frac{\text{Final Vessel Mass - Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.16.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.18).

11.17. Micro-concentration (Turbovap)

Concentrate the extracts in 35 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 50-35 mL culture tubes. Other turbo-evaporator models can be used that may or may not have the same culture tube sizes and/or capacity. Adjust temperature according to solvent (65ºC for toluene and 50ºC for hexane or hexane/ methlyene chloride mixtures)

11.17.1. The evaporating times are dependent on sample volume and solvent. The following are examples and can change from sample to sample. Each sample should be checked in intermittent intervals to make sure samples do not go dry.

11.17.2. When evaporating 30 mL toluene, it will normally take approximately 30-50 minutes with the temperature setting described above.

11.17.3. When evaporating 30 mL hexane/ methlyene chloride, it will normally take approximately 10-20 minutes with the temperature setting described above.

11.17.4. For samples requiring % Lipids analysis:

11.17.4.1. Evaporate to near dryness. Add approximately 5 mL hexane and concentrate to ensure that only the lipids are present.

11.17.4.2. Dry the concentration vessel and let stand at room temperature. Weigh the vessel and record on the benchsheet.

11.17.4.3. Calculate % lipids as follows:

$$\% \text{ Lipids} = \frac{\text{Final Vessel Mass - Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.17.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.18).

11.18. Recovery Standard

11.18.1. Transfer extracts to a micro concentration vial (test tubes and other small vessels may also be used)

11.18.2. With a stream of dry, purified nitrogen, reduce the extract volume to

approximately 100 µL.

11.18.3. Add 20 µL of the recovery standard solution (Table 2).

11.18.4. With a stream of dry, purified nitrogen, reduce the extract volume to 20 µL.

11.18.5. Transfer the extract to an autoinjection vial and store in the dark at room temperature.

11.18.6. A smaller final volume can be used to decrease the detection limit upon client approval.

11.18.7. A larger final volume can be use to decrease potential matrix interferences, if the column and acid cleanups were unsuccessful.

11.19. Sample Dilution Procedure

11.19.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$\text{Final Conc. of Extract} = \frac{(\text{Conc. of original extract}) \text{ x } (\text{Amount of aliquot taken})}{(\text{Volume of diluted extract})}$$

Ex:
$$\frac{(10\,\text{g}) \text{ x } (2\,\mu\text{L})}{(20\,\mu\text{L}) \text{ x } (100\,\mu\text{L})} = \frac{1\,\text{g}}{100\,\mu\text{L}} \text{ FV}$$

Record the final sample concentration on the extract label.

11.19.2. Complex dilution requiring respiking of IS and RS:

Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL RS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. .

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.2. Toluene, which is a less hazardous solvent, has been substituted for benzene as an extraction solvent.

14.3. The use of SoxTherm extraction rather than soxhlet extraction, when appropriate, reduces the volume of solvent used.

14.4. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.5. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.6. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.7. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials, silica gel, alumina, and carbon from column clean-ups, contaminated with various methylene chloride, solvents and eluates. Dump the materials into a contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Flammable solvent and methylene chloride waste generated during glassware and sodium sulfate cleaning. Solvent waste collected during roto-vap/turbo-vap reduction

of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Assorted flammable solvents and methylene chloride waste generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing.. Waste solvents and methylene chloride collected during roto-rap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.6. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Deviations from EPA 8290 and 8290A.

17.1.1. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.1.2. Extract clean-ups are performed at the discretion of the analyst when interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.

17.1.3. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.

17.1.4. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any mechanical means to break up an emulsion.

17.1.5. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.

17.1.6. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2, 8290A Section 7.3.6.).

17.2. Modifications from TO-9A method

17.2.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.

17.2.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.

17.2.3. The $^{37}Cl_4$-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.4. Samples are extracted with toluene not benzene.

17.2.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

17.2.6. All cleanup procedures are optional and applied based on the analyst's discretion.

17.2.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.2.8. The final volume is adjusted to 20 µL in tetradecane.

17.2.9. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of Sample Fortification and Recovery Standard Solutions.

18.3. Table 3 - The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Figure 1 - Analysis Flowchart

18.5. Figure 2 — IFB column cleanup

18.6. Figure 3 — D2 Column cleanup

18.7. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-IDP-0005, Revision 1.1, Effective 2/12/2010

19.1.1. Section 11.2 – updated SOP reference from SAC-ID-0009 to WS-ID-0009.

19.1.2. Section 11.6.1 – changed: "Prior to loading samples, run the system through

a cleaning cycle (approximately 3 hours)" to "(approximately 1 hour)."

19.1.3. Section 11.6.8 – changed "…fresh charge (140 mL) of toluene…" to "…fresh charge (150 mL) of toluene….".

19.1.4. Section 11.16.1 – inserted in Table "No vacuum applied" under vacuum setting (PSI) for solvent Methylene chloride.

19.2. WS-IDP-0005, Revision 1, Effective 10/2/2008

19.2.1. Added 8290A references.

19.2.1.1. Extract and standard storage.

19.2.1.2. Removal of MS/MSD.

19.2.2. Updated to TestAmerica format.

19.2.3. Separated the analytical steps from the preparation steps, this SOP is concerned only with the sample preparation.

19.3. WS-ID-0005, Revision 6.7, Effective 8/21/2008

19.3.1. Changed the word "toluene" to "acetone" in 7.11.2.

19.4. WS-ID-0005, Revision 6.6, Effective 4/9/2008

19.4.1. Added South Carolina rule to prepare an MS/MSD with every batch.

19.4.2. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (μL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Recovery Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/µL; Solvent: Isooctane	Recovery Standard Solution Concentration pg/µL; Solvent: Tetradecane
$^{13}C_{12}$ -2,3,7,8-TCDD	2[a], 100[c]	--
$^{13}C_{12}$ -2,3,7,8-TCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4-TCDD	--	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDD	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8-PeCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8,9-HxCDD	--	100
$^{13}C_{12}$-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
$^{13}C_{12}$ -2,3,4,7,8-PeCDF[c]	100[c]	
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
$^{13}C_{12}$ -OCDD	4[a], 200[c]	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) $^{13}C_{12}$ -1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and $^{13}C_{12}$-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an internal standard.
(+)The ^{13}C -labeled analog is used as a recovery standard.

FIGURE 1 — Flowchart of Process

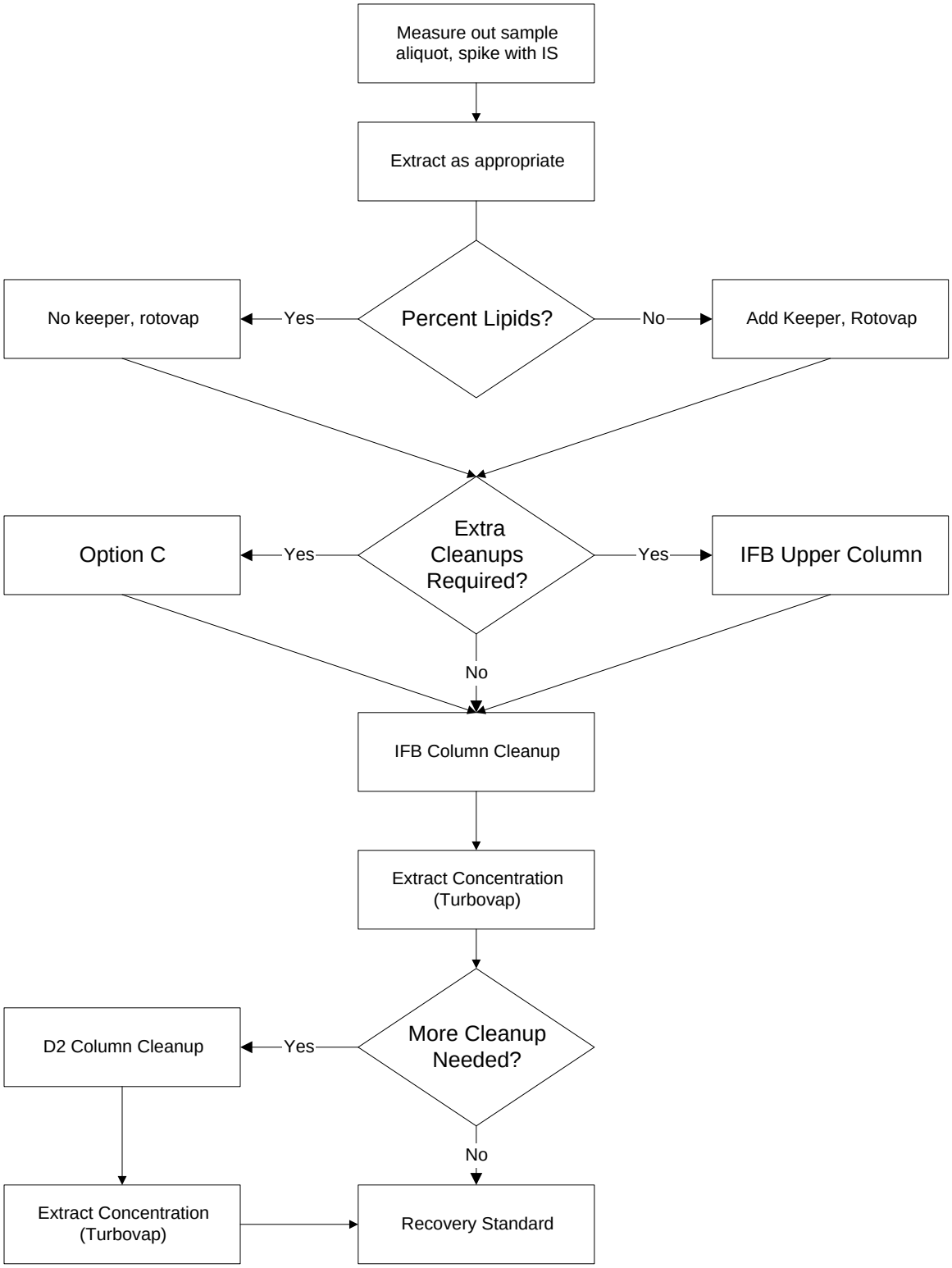


Figure 2 – Diagram of IFB Column Cleanup

Use 20 mm column for top column (IFB Column)
Use 16 mm column for bottom column* (Acid Alumina)
Note: Upper and lower columns are piggy backed for IFB cleanup, upper column only can be used for additional cleaning.

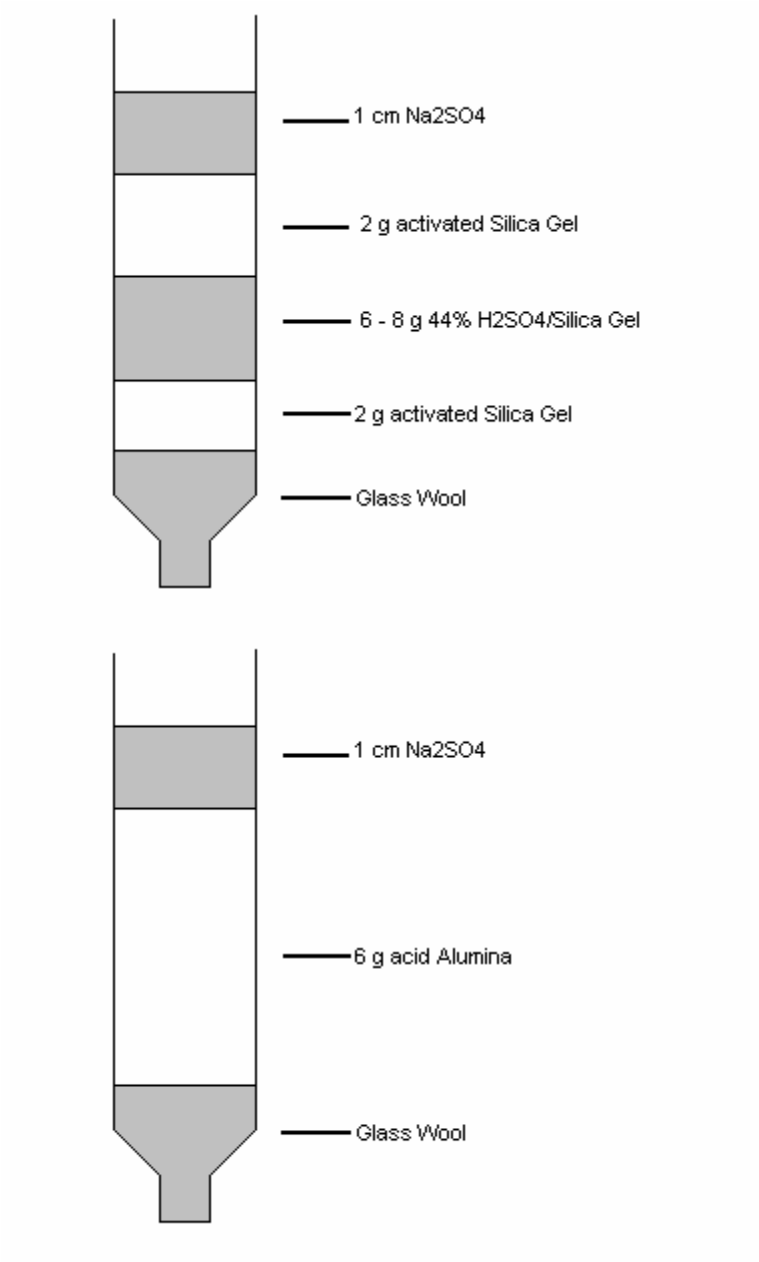
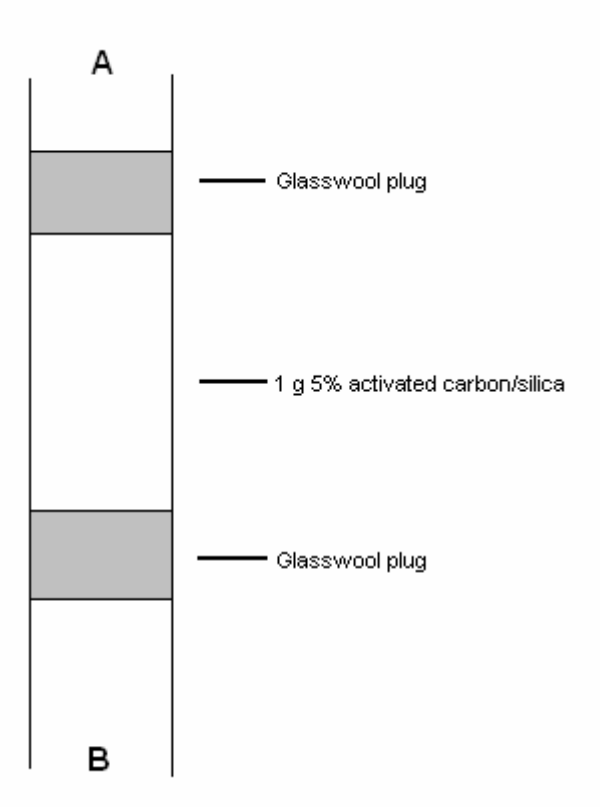


Figure 3— D2 Carbon Column:



APPENDIX A — Screening the Laboratory for 2,3,7,8 Congeners

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST
Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION
Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS
Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT
Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS
Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION
An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency

particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

SOP Change in Progress Attachment (CIPA)

SOP Number	SOP Title	SOP Revision	SOP Effective Date	CIPA Effective Date
BR-WC-024	TOC in Soil	0	05/10/11	05/10/11

The following revisions were made to this standard operating procedure (SOP). These changes are effective as of the CIPA Effective Date. Changes to this document will be incorporated into the document with the next revision. This document change is authorized and issued by the laboratory's QA Department.

Section 7.2: Add the following text to this section:

- Potassium Hydrogen Phthalate (KHP) (Primary Standard Grade) Used to calibrate the instrument. 47.05% Carbon by weight

1% Carbon KHP Solution (10,000 mg Carbon/L): Add 50 mL of reagent water to a 100 mL volumetric flask. Add 2.128 g of KHP and dissolve completely. Adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration of 6 months from the date prepared and store at room temperature.

0.1% Carbon KHP Solution (1000mg Carbon/L): Add approximately 25 mL of reagent water to a 50 mL volumetric flask. Add 5 mL of 1 % Carbon KHP solution to the flask and adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration date of 6 months from the date prepared so long as the parent solution does not expire sooner, in which case use the earliest expiration date. Store the solution at room temperature.

0.01% Carbon KHP Solution (100mg Carbon/L): Add approximately 25 mL of reagent water to a 50 mL volumetric flask. Add 0.5 mL of 1% Carbon KHP Solution and adjust to final volume with reagent water. To mix the solution, cap the flask and invert. Allow the air bubble to reach the top of the flask. Repeat 9 times. Assign an expiration date of 6 months from the date prepared so long as the parent solution does not expire sooner, in which case use the earliest expiration date. Store the solution at room temperature.

Section 9.1: Change the LCS and MS acceptance criteria to 75-125%R.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	%R ~~85-115~~ 75-125
Sample Duplicate (DP)	Client Request	RPD (≤ 20)
Matrix Spike (MS)	Client Request	%R ~~85-115~~ 75-125

Section 10.1: Replace the table in this section with the following table:

Calibration Standards	1.0% C KHP uL	0.1% C KHP uL	0.01%C KHP uL	% Carbon KHP	Carbon (mg)	mg/Kg of Carbon (10mg sample)
Level 1	0	0	0	47.05	0	0
Level 2	0	0	100	47.05	0.010	1000
Level 3	0	40	0	47.05	0.040	4000
Level 4	25	0	0	47.05	0.25	25000
Level 5	50	0	0	47.05	0.500	50000
Level 6	75	0	0	47.05	1.000	75000

Title: TOC in Soil

Approval Signatures:

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Approval Date: May 10, 2011

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) and black carbon in soils, sediments and other solids.

The procedure for TOC in soils and sediments is provided in the main body of this SOP. The procedure for the determination of TOC in marine sediment high in inorganic carbon is provided in Appendix B and the procedure for black carbon is provided in Appendix D.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used to determine percent dry weight in soil and solid materials.

The routine reporting limit is 1000 mg/kg based on an initial sample weight of 10 mg. Additional weight of sample may be used (up to 25 mg) to achieve as low a reporting limit as 500 mg/kg.

2.0 Summary of Method

A 10 mg aliquot of sample is transferred to a tin capsule, treated with phosphoric acid and dried in an oven at a temperature 105°C for 30 minutes to one hour in order to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is analyzed on an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

This procedure is based on the following reference documents:

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.

The procedure in this SOP for total organic carbon is modified from the above reference method. The procedures for black carbon and marine sediment are not based on a method and should be considered laboratory derived methods.

3.0 Definitions

A list of general laboratory terms and definitions are provided in Appendix A.

4.0 Interferences

Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples

and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure. A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory's Environmental Health and Safety Coordinator.

6.0 Equipment and Supplies

- Drying Oven: Capable of maintaining a temperature of 105 ±2°C.
- Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.
- Costech Elemental Analyzer: Model 4010 or equivalent.
- Analytical Balance: Capable of weighing to the nearest 0.001mg.
- Aluminum Weigh Boats.
- Tweezers
- 5mm X 9mm tin capsules
- Quartz Columns: Costech Analytical or equivalent.
- Quartz wool: for segregating and containing column materials
- Copper Wire, Reduced: Costech Analytical or equivalent.
- Tungsten on Alumina: Costech Analytical or equivalent.
- High Temperature Gloves
- Clear Plastic Sample Trays: Costech Analytical or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Reagent water

- Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration date of six months from date made and store the solution at room temperature.

7.2 Standards

- Acetanilide Crystals of known Carbon percentage: Purchased from Costech Analytical. Used to check instrument calibration.

- Sulfanilamide Crystals (41.84% Carbon): Purchased from Costech Analytical. This material is used to calibrate the instruments.

- Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.

- Matrix Spike Material, 1632B trace elements in coal (76.86% Carbon)

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so sampling procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the laboratory recommended minimum sample size, preservation and holding time requirements:

Parameter	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Total Organic Carbon	Amber glass	10 g	Chilled to ≤ 4°C	14 Days	TOC by Lloyd Kahn
Black Carbon	Amber glass	10 g	Chilled to ≤ 4°C	None	None

[1] Holding time is determined from date of collection.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance

		Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	%R (85-115)
Sample Duplicate (DP)	Client Request	RPD (≤ 20)
Matrix Spikes (MS)	Client Request	%R (85-115)

9.2 Instrument QC

The laboratory analyzes the following instrument check standards:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initial Method Set-Up, after combustion chamber is changed (approx. every 200 drops)	Correlation coefficient must be >0.995
Calibration Verification (Acetanilide)	Every 20 drops and at the end of the analytical sequence	%R (85-115)
Calibration Blank (CCB)	After every acetanilide	<RL

10.0 Procedure

10.1 Calibration

Analyze a calibration curve each time the combustion column is changed. Change the column after 200 drops or when you experience result issues or odd peak shapes or baseline issues. The column change procedure is provided in Appendix C.

The recommended formulations for each calibration level are provided in the following table:

Calibration Standard Sulfanimide	Weight[1] (mg)	% Carbon	Carbon (mg)
Calibration Level 1	0.100	41.84	0.0418
Calibration Level 2	0.500	41.84	0.2092
Calibration Level 3	1.00	41.84	0.4184
Calibration Level 4	1.50	41.84	0.6276
Calibration Level 5	1.75	41.84	0.7322

[1]These weights are approximate. Enter the actual weight used into the software program.

Measure a single drop for each calibration point. The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to further analysis.

1.0 Troubleshooting

- Calibration passes at > 0.995 correlation, but LCS fails abnormally low: Re-calibrate. Calibration usually needs to be > 0.999 correlation.

- Carbon peak “maxes out” at instrument 1200mv (peak has flat top): Reanalyze sample at lower weight.

- No peaks on any chromatograms, no results: Gases to instrument may be off. Turn on all gasses at valve manifold.

- Autosampler will not work at all: Gasses to instrument may be off. Turn on all gasses at valve manifold.

- Single chromatogram shows results at bottom of page, but no peak or baseline in chromatogram window: Re-print single chromatogram.

- Some or all chromatograms show carbon peak at same retention time as Acetanilide, but peak is not identified as carbon, or is identified as another element: Retention time shifted. Adjust retention time in calibration window, and reprint chromatograms.
- Upon recalibration, peaks are not being identified as carbon: In calibration window, general tab, adjust retention time to match peaks. Starting at level 1, “Open Standard”, open level1 curve pt. in calibration directory, click “Add Peak” button, click on peak itself. Increase level #, opening standard for each curve pt and add each peak. Carbon Tab should have all five calibration points on curve, if done correctly.
- Peaks in chromatograms identified as carbon, but all results in summary table below chromatogram are zero: Current calibration not associated with run when started. Open current calibration, copy first two columns for all points (5 rows) in small table in general tab. Then, open calibration that was associated with run (should be empty) and paste into table in calibration tab. Reprint all chromatograms on run.
- Software crashes during analysis: Boot up software normally. Chromatograms already printed/analyzed are ok, but, sample that was analyzing during shutdown is lost. Restart table at next sample by un-checking “run” box for samples already run and sample that was lost.

- Autosampler error causes few samples to remain in autosampler tray after run has finished: Identify samples that got stuck. Create a new run and analyze stuck samples (with initial weights) with bracketing QC. No PBS/LCS needed.
- Autosampler error causes many sequential samples to remain in autosampler tray after run has finished (usually end of run): Add rows onto existing table. Identify samples that did not get analyzed and repeat Ids and weights into added rows. Restart table. All analyzed samples’ status should be blue (analyzed), added rows should be green (not analyzed yet).
- Various result issues or odd peak shapes or baseline issues: Column may be leaking or cracked. Change column, recalibrate.

10.3 Sample Preparation

Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare the balance. Using the small sample scoop, add approximately 10 mg (or the project specified sample weight) of sample to the capsule. Record the actual sample weight used on sample preparation log. Remove the capsule from the balance and place into one of the aluminum holding trays. Weigh two additional portions of sample into two separate tin capsules for each field sample.

To prepare the method blank, set two empty tin capsules into an aluminum holding tray.

To prepare the LCC, weigh ~9 mg of the LECO LCS material into two separate tin capsules and set them in sequence in an aluminum holding tray.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 0.3 – 0.7 mg of matrix spike material and record this weight.

For the sample duplicate, weigh out an additional sample aliquot. Prepare two aliquots for both the matrix spike and the sample duplicate.

Add two drops of 1:19 phosphoric acid to each tin capsule. Place the aluminum trays into a drying oven set to a temperature of 105ºC for 30-60 minutes or until all samples appear dry.

Using tweezers pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the acetanilide check standards and associated calibration blanks.

Prepare the acetanilide standard and blanks as follows:

For each acetanilide spike, weigh ~0.5 mg of acetanilide material into a tin capsule. Fold the capsule up and compress down to the smallest size possible. Prepare enough acetanilide to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the autosampler tray.

Software Set-up and Analysis

If the column has been changed generate a new calibration curve. If not, use the existing calibration curve for analysis. Each column will analyze approximately 200 individual sample drops. When the counter on the instrument approaches 200, watch the instrument data for signs that the column is deteriorating; poor peak resolution, trailing baselines, extraneous peaks. If a column change is necessary, refer to Appendix C for the procedure. After changing the column, generate a new calibration curve.

Select the appropriate channel: Channel 1 is the NA 1500, Channel 2 is the EA 1108, and Channel 3 is the Costech instrument, which has its own PC. At the main screen select the sample table icon. The last sample table that was run will be shown on the screen.

Open a new sample table, and select the appropriate number of sample positions for the analysis, then name the table with the date and a unique alpha designator (i.e. 061505a). In front of the %3r in the file name column of the sample table, add the sample table name to ensure that each individual chromatogram generated from this sample table has a unique filename associated with it.

If the combustion column has been changed and instrument needs to be calibrated, follow the procedure below:

Prepare a “bypass” drop to determine the retention time for carbon with the new column. The bypass is an aliquot of acetanilide. The weight is not needed. Drop the bypass into the

instrument and initiate a singular analysis. Set the retention time for carbon in the software to match that of the bypass drop.

Identify the first five sample lines with the names Std1 through Std 5. Enter their respective weights in the weight column, assign them a level # in the level column (Std1 is level 1, Std2 is level 2, etc.) to alert the software the order in which to place the calibration standards. In the sample type column, use the drop down and select "standard" for each. Finally, use the drop down in the Standard name column and select "sulfanilamide" for each. Add the standards to the autosampler tray and hit "start" to run the calibration.

Sample Analysis:

Open a new sample tray and create a unique file name. When the instrument was last calibrated, the software creates a calibration file with the same name as the sample table in which it was run. Open this file and save it with the same name as the sample table about to be run to ensure that the analysis is calculated from the most recent calibration. To do this, click on the calibration icon (looks like a little calibration curve) and use the file option to open the calibration file last performed. Save this file with the same name as your sample table. Click on the sample table icon (looks like a little sample table) to get back to your sample table.

Enter each sample ID and their respective weights and save the sample table. Enter a weight of 10 mg for the Method Blank (PBS) and instrument blanks.

An example analytical sequence follows:

Initial Calibration (calibration blank and 5 calibration standards)

Acetanilide	(1 drop)
Blank	(1 drop)
PBS	(2 individual drops)
LCS	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Acetanilide	(1 drop)
Blank	(1 drop)

Add the samples and acetanilides to the autosampler tray and set the tray into the autosampler carriage. Turn the autosampler tray until the number 1 position is behind the post, in front of the autosampler. The tray is now set to run.

Click the "start" icon to begin the analysis.

After analysis review the analytical results against the acceptance criteria given in Table 2, Section 18.0, and perform corrective action as necessary. Report results in mg/kg Carbon and corrected for % solids.

11.0 Calculations / Data Reduction

11.1 Calculations

11.2 Percent Carbon to mg/kg Carbon Conversion

% Carbon × 10,000 = mg/kg Carbon

11.3 LCS Percent Recovery (%R)

$$\%R = \frac{\text{LCS Result}}{\text{LCS True Value}} \times 100$$

11.4 MS Percent Recovery (%R)

$$\text{mg/Kg wet SA} = \frac{\text{Spike TV} \times \text{weight of MS added}}{\text{sample weight}} \times 1\text{million}$$

$$\text{mg/Kg dry SA} = \frac{\text{mg/Kg wet SA}}{\%\,\text{solid}} \times 100$$

$$\text{mg/Kg dry Carbon} = \frac{\text{mg/Kg wet Carbon (from instrument)}}{\%\,\text{solid}} \times 100$$

$$\%R = \frac{A - B}{C} \times 100$$

Where:
A= Average of two drops of MS sample result: mg/Kg dry carbon
B= Average of two drops of parent sample: mg/Kg dry carbon
C= Average of two drops of mg/Kg dry SA
SA= spike added (mg/Kg)
Spike TV= 0.7686 (mg/Kg)

11.5 Relative Percent Difference (RPD)

$$RPD = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

11.6 Dixon Test (Use 3-7 results)

1. Sort all the results in ascending order (low values to high).
2. Calculate the tau statistic for the low and high values.
3. Compare the calculated tau statistics (low and high) to critical values listed below.
4. If either calculated tau is higher than the critical value, reject that value and repeat the test.

Tau statistic for lowest value = $\tau_L = (X_2 - X_1) / (X_k - X_1)$
Tau statistic for highest value = $\tau_H = (X_k - X_{k-1}) / (X_k - X_1)$

Where:
X_2 = Second lowest value in sorted list.
X_1 = Lowest value in sorted list.
X_k = Highest value in sorted list.
X_{k-1} = Second highest value in sorted list.

Number of observations, k	Critical Values
3	0.941
4	0.765
5	0.642
6	0.560
7	0.507

11.2 Data Review

12.0 Method Performance

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out.

- Caustic waste – 2.5 L glass satellite container.
- Acidic Waste - 2.5L glass satellite container

The satellite containers are labeled “Hazardous Waste” along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations.

15.0 References / Cross-References

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.
- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.
- Corporate SOP CW-E-M-001 Corporate Environmental Health and Safety Manual
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-011 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

The laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	TOC by Lloyd Kahn	The laboratory analyzes two drops per sample and if the RPD is greater than 40% the Dixon test is utilized.

17.0 Attachments

- Table 1: Primary Materials Used
- Table 2: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: TOC Procedure for High Concentration Marine Sediments (CITHON)
- Appendix C: Column change procedure
- Appendix D: Determination of Black Carbon in Sediment Procedure

18.0 Revision History

BR-WC-0024, Revision 0:

This is the first version of this SOP.

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Following each column change	correlation coefficient ≥ 0.995	Standards check, re-calibration
Acetanilide	Every 20 drops and at the end of the analytical run	%R (85-115)	Re-prepare and reanalyze samples not bracketed by passing standard.
Blank (paired with Acetanilide)	Following each Acetanilide	< RL	Re-prepare and reanalyze batch.
Method Blank (MB)	Once per batch of 20 samples	< RL DoD: ½ RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples	%R (75-125)	Re-prepare and reanalyze batch.
Sample Duplicate (DP)	One per batch of 20 or less samples	RPD (≤ 20)	Discuss outlier in project narrative
MS/MSD	One per batch of 20 or less samples	%R (75-125)	Discuss outlier in project narrative
Sample precsion	Each sample is run in duplicate	%RPD<40%	Analyze 2 more replicates and perform Dixon test for high and low outliers. Include Dixon spreadsheet in the data package and narrative note results.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration. A standard containing the analyte in question (sulphanilimide) is prepared at varying weights and analyzed. This standard is a separate source from the LCS. The sulphanilimide is used to calibrate the instrument response with respect to analyte concentration.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Appendix B: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle. Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!*** Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample "boils over" it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid in a 105-degree oven.

Dry the treated samples in the oven after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

NOTE*: Samples are hydroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

NOTE: *Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix C: Column Change Procedure

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don't over tighten.

Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to

1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

Appendix D: Determination of Black Carbon in Sediment Procedure

1. Obtain a representative subsample of the sediment. Weight 10 grams of sample into a clean pre-tared aluminum drying pan or equivalent.

2. Dry the sample at 105^{o}C for at least 12 hours.

3. Grind the sample using a mortar and pestle.

4. Sieve the sample using a number 35 sieve (500 um).

5. Treat the sample with phosphoric acid. Add acid drop wise until effervescence is no longer observed.

6. Dry the sample at 105^{o}C for 1 hour.

7. Set aside an aliquot of the sample at this stage for direct TOC analysis, reported without correction for the IN623 percent solids. Continue with the sample for Black Carbon.

8. Place the dried sample into a clean crucible and cover the sample.

9. Bake the samples at 375^{o}C in a muffle for 24 hours or until the LCS is +/- 50% of the true value.

10. Allow the samples to cool and transfer approximately 5.0 mg into each of two tin capsules.

11. Transfer the sample (in the tin capsules) to the TOC analyzer for analysis by the Lloyd Kahn Method.

12. The sample is pyrolyzed in an inductive type furnace, where the carbon is converted to carbon dioxide, which is measured using a differential thermal conductivity detector.

13. The results will be reported as mg/Kg Black Carbon.

Note: Black carbon LCS material: NIST Standard Reference Material 1944 New York-New Jersey Waterways Sediment.

References:

Orjan Gustafsson, Thomas D. Bucherli, Zofia Kukulska, Mette Andersson, Claude Largeau, Jean-Noel Rouzaud, Christopher M. Reddy and Timothy I. Eglinton (December 2001) Evaluation of a Protocol for the Quantification of Black Carbon in Sediments, Global Biogeochemical Cycles, Volume 15, pages 881-890.

Orjan Gustafsson, Farnaz Haghseta, Charmaine Chan, John MacFarlane & Philip M. Gschwend (1997) Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability, Environmental Science & Technology, Volume 31, pages 203-209.

Quality Assurance Manual

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Quality Manager - John P. Morris	Date 07/15/11
Technical Director – Michael Phillips	Date 07/15/11

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REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

REFERENCED LABORATORY SOPs

SOP Reference	Title
DV-QA-0010	Document Control & Updating Procedures (Sec. 3.4.1)
DV-QA-013P	Customer Complaints (Sec .10.1)
DV-QA-028P	Method of Change(Sec. 3.4.1; 13.2)
DV-QA-0005	Document Archiving Procedure (Sec. 14.1.4)
DV-QA-0024	Training (Sec. 17.3)
DV-QA-001P,	Preparation and Management of Standard Operating Procedures (Sec. 19.2)
DV-QA-0024	Training (Sec. 19.4.2)
DV-QA-005P	Determination of Method Detection Limits (Sec. 19.7)
DV-QA-017P	Electronic Reporting (Sec. 19.14.1)
DV-QA-0023	Subsampling (Sec. 22.5)
DV-QA-0003	Sample Management and Chain of Custody (Sec. 23.2.1.3)
DV-QA-003P	Quality Assurance Program (Sec. 5.5, 23.2.1)
DV-QA-0010	Document Control (Sec. 6.1)
DV-QA-001P	Preparation and Management of Standard Operating Procedures and Other Controlled Documents (Sec. 6.3)
DV-QA-0005	Document Archiving Procedure (Sec. 6.4)
DV-QA-011P	Acceptable Manual Integration Practices (Sec. 19.14.2)
DV-QA-025P	Electronic Data Backup (Sec. 14.1.3)

Section references are to the first occurrences only.

SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

TestAmerica Denver Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E) In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs

listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *U.S. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th–21st, and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 3.6, November 2010.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP),* Version 4.0.02, May 2006.
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. DV-QA-0010).

SECTION 4. MANAGEMENT REQUIREMENTS

4.1 Overview

TestAmerica Denver is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Denver is presented in Figure 4-1.

4.2 Roles and Responsibilities

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Additional Requirements for Laboratories

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Denver laboratory.

4.2.2 Laboratory Director

TestAmerica Denver's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical directors for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing. The role of the Technical Director at TestAmerica Denver is fulfilled by the Laboratory Director or appointed designee(s).

- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Technical Director(s), and the Operations Manager as direct reports.

4.2.3 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.

- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025.

4.2.4 Quality Assurance Specialist

The Quality Assurance Specialist performs several roles. The QA Specialist reports to the facility QA Manager. The QA Specialist is responsible for QA documentation and involvement in the following activities:

- Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.
- Facilitate external audits, coordinating with the QA Manager and Laboratory Staff to address any deficiencies noted at the time of the audit and subsequently presented in the final audit report.
- Assist the QA Manager in the preparation of new SOP's and in the maintenance of existing SOPs, coordinating annual reviews and updates.
- Manages the performance testing (PT) studies, coordinates follow up studies for failed analytes and works with QA Manager and Laboratory Staff to complete needed corrective action reports.
- Personnel training records review and maintenance.
- Document control maintenance.
- Assists the Quality Manager and Project Management Group in the review of program plans for consistency with organizational and contractual requirements. Summarize and convey to appropriate personnel anomalies or inconsistencies observed in the review process.
- Manages certifications and accreditations.
- Monitors for compliance the following QA Metrics: Temperature Monitoring of refrigeration units and incubators; thermometer calibrations; balance calibrations; eppendorf/pipette calibrations; and proper standard/reagent storage.
- Periodic checks on the proper use and review of instrument logs.
- Initiate the Mint-miner data file review process for organic instrumentation. Maintain tracking sheet of activity.
- Initiate the annual Instrument review.
- Assist in the technical review of data packages which require QA review.

4.2.5 Quality Assurance Assistant

The Quality Assurance Assistant performs several roles. The QA Assistant reports to the facility QA Manager. The QA Assistant is responsible for QA documentation and involvement in the following activities:

- Assist the QA Manager in performing the annual internal laboratory audits, compiling the evaluation, and coordinating the development of an action plan to address any deficiency identified.

- Serves as a project manager for proficiency testing samples and other QC samples. Processes and reports QC samples as routine samples to appropriate agencies.
- Assist the QA Manager in maintaining the laboratory's reference data to keep it current and accurate.
- Prepares certification applications for states as directed by QA Manager.
- Personnel training records review and maintenance.
- Document control maintenance.
- Assisting departments in generating MDL spreadsheets and calculations, reviewing MDL studies submitted to QA.
- Assisting in control limit generation.
- Ensuring maintenance of records archives.
- Maintaining historical indices for all technical records including SOPs, QC records, laboratory data, etc.

4.2.6 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Operations Manager. He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results.
- Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data

quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.

- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.

4.2.7 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. He/She reports directly to the Laboratory Director. He/She acts as the Technical Director in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.8 Radiation Safety Officer

The Radiation Safety Officer (RSO) is responsible for implementing TestAmerica Denver's radiation safety program. The RSO reports directly to the Technical Director. The RSO's duties consist of:

- Manage the personnel radiation dosimetry program
- Maintains the Radioactive Materials License and radionuclide inventory

- Monitors laboratory operation for compliance with the Radiation Safety Manual
- Training, documenting, and evaluating the TestAmerica Denver personnel for handling radioactive material
- Creating, releasing, and decontaminating of Radiological Control Areas (RCAs)
- Monitoring and tracking of radioactive materials
- Conducting the radioactive material waste disposal program in accordance with State and Federal regulations
- Maintaining all records related to the radiation safety program

4.2.9 Employee Health and Safety Coordinator

The EH&S Coordinator is responsible for administering the EH&S program that provides a safe, healthy working environment for all employees and the environment. The Employee Health and Safety Coordinator (EH&S Coordinator) reports directly to the Laboratory Director and the corporate Environmental Health and Safety Director. He/She monitors all areas for unsafe conditions, acts, and potential hazards. Specific responsibilities include, but are not limited to:

- Staying current with the hazardous waste regulations
- Continuing training on hazardous waste issues
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.

- Determine when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

4.2.10 Hazardous Waste Specialist

The Hazardous Waste Specialist is responsible for coordinating and implementing the divisional hazardous waste program to ensure compliance with all federal, state, local laws, and company policies. The Hazardous waste specialist reports to the EH&S Coordinator. The duties consist of:

- Staying current with the hazardous waste regulations
- Conducts weekly inspections of satellite accumulation areas and all hazardous waste storage areas
- Operates and maintains on-site wastewater treatment system
- Coordinates the proper storage, packing and disposal of laboratory wastes according to Department of Transportation (DOT) and Resource Conservation and Recovery Act (RCRA) regulations
- Maintains waste disposal records
- Coordinates spill response activities including documentation for waste storage areas

4.2.11 Waste Disposal Technician

The Waste Disposal Technician is responsible for proper disposal of spent chemicals, process waste, and unused laboratory samples used in the laboratory according to corporate, federal, state, and local guidelines. The Waste Disposal Technician reports to the Hazardous Waste Specialist and EH&S Coordinator. The duties consist of:

- Packaging hazardous waste for transport per DOT, RCRA and TSCA guidelines
- Identifying waste streams and maintaining satellite accumulation areas
- Packages expired chemicals for shipment or disposal
- Tracks volume of waste generated for reporting to corporate and EPA
- Prepares and tracks implementation of the Waste Minimization Plan
- Empties satellite containers into bulk containers and returns to the laboratory for reuse

4.2.12 Department Manager

Department Managers report to the Operations Manager. At TestAmerica Denver there are two levels of Department Managers (I or II). The level designation is based on the level of experience. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in

compliance and if new, modified, and optimized measures are feasible and should be added to these documents.

- With regard to analysts, participates in the selection, training, development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He/She is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.13 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The Analyst position at TestAmerica Denver is divided into levels. These levels range from Analyst I to Analyst V. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.14 Laboratory Technician

Laboratory Technicians are responsible for the preparation of samples and performing all tasks assigned to them by the group leader or supervisor. The Laboratory Technician position at TestAmerica Denver is divided into three levels. These levels are Laboratory Technician I, Laboratory Technician II, and Laboratory Technician III. The level designation is based on experience, expertise, and responsibilities. The responsibilities of the Laboratory Technician are listed below:

- Retrieving samples from Sample Control for analysis
- Performing sample preparation by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Documenting standard and sample preparation, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database
- Report all non-conformance situations, sample preparation problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

4.2.15 Laboratory Assistant

The Laboratory Assistant position is an entry-level position to learn basic laboratory technician skills. The Laboratory Assistant reports to their group leader or supervisor. The Laboratory Assistants duties include the following:

- Assisting the Laboratory Technicians in preparation of samples for analysis
- Preparing routine forms and reports
- Collecting and preparing materials and supplies for the laboratory
- Assisting technicians in conducting routine analysis.

4.2.16 Sample Control Manager

The Sample Control Manager reports to the Project Management Manager. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS
- Ensure the verification of data entry from login
- Provide daily assessments of sample receipts
- Monitor the preparation and shipment of bottle kits to clients
- Oversee the receipt, log in, and storage of samples
- Schedules couriers for sample pickup from customer sites

4.2.17 Sample Control Technician

The Sample Control Technician reports to the Sample Control Manager. The Sample Control Technician position at TestAmerica Denver is divided into levels. These levels range from Sample Control Technician I to Sample Control Technician IV. The level designation is based on experience and responsibilities of the Technician. The Sample Control Technician responsibilities include the following:

- Receive and unload samples or consignments in accordance with DOT regulations
- Verify samples against the Chain of Custody (COC)
- Log in sample into the LIMS to assign a lot number for tracking purposes and distribute the paperwork to the Project Managers and Department Managers
- Label samples with lot number assigned and deliver the samples to the appropriate labs for analysis daily
- Monitor freezer and cooler temperatures daily to confirm that the readings are within SOP guidelines
- Ship all subcontracted samples to designated lab in accordance with DOT regulations as needed

4.2.18 Shipping/Maintenance Technician

The Shipping/Maintenance Technician reports to the Sample Control Manager and the Project Management Manager. The Shipping/Maintenance Technician duties include the following:

- Maintaining the inventory control system
- Receiving and distributing incoming supplies
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Maintaining bottle and cooler inventory
- Packing in-house samples for shipment to other laboratories

4.2.19 Courier

The Courier reports to the Sample Control Manager and the Project Management Manager. The Courier's duties include the following:

- Picking up and delivering samples and reports to clients and the laboratory
- Receiving and signing the chain of custody for samples
- Preparing and shipping bottle sampling kits to clients or on-site crews
- Performing preventative maintenance on company vehicles

4.2.20 Project Management Manager

The Project Management Manager reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team
- Technical liaison for the Project Management team
- Human resource management of the Project Management team
- Responsible to ensure that clients receive the proper sampling supplies
- Accountable for response to client inquiries concerning sample status
- Responsible for assistance to clients regarding the resolution of problems concerning COC
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory
- Notifying the supervisors of incoming projects and sample delivery schedules
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff

- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports
- Inform clients of data package-related problems and resolve service issues
- Coordinate requests for sample containers and other services (data packages)

4.2.21 Project Manager

The Project Managers report to the Project Management Manager and serve as liaisons between the laboratory and its clients. At TestAmerica Denver there are two levels of Project Managers (I or II). The level designation is based on experience, expertise, and responsibilities. The Project Manager's responsibilities include:

- Ensuring client specifications are met by communicating project and quality assurance requirements to the laboratory
- Notifying laboratory personnel of incoming projects and sample delivery schedules
- Monitoring the status of all projects in-house to ensure timely delivery of reports
- Informing clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff
- Coordinating client requests for sample containers and other services
- Scheduling sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinating subcontract work
- Assisting clients in procuring the proper sampling supplies
- Responding to client inquiries concerning sample status
- Assisting clients with resolution of problems concerning Chains-of-Custody

4.2.22 Project Management Assistant

The Project Management Assistant reports to the Project Management Manager and designated Project Manager. The Project Management Assistant assists the Project Manager in servicing the client's needs and communicating those needs to the laboratory. The Project Management Assistant's responsibilities include:

- Collating data reports, expanded deliverables, CLP data packages and electronic data deliverables (EDD's) for delivery to clients.
- Writing case narratives accompanying data packages to communicate anomalies to clients
- Entering data from subcontracted laboratories
- Proof reading and filing data reports received from the laboratory

- Assisting Project Managers in changing compound lists, TAT, and setting up tables in Word or Excel
- Monitoring report due dates for timely delivery
- Invoicing completed data packages
- Generating credit or debit invoices to ensure proper payment
- Copying and paginating reports

4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Robert C. Hanisch Laboratory Director	Brett VanDelinder Customer Service Manager
John Morris Quality Manager	Peggy Sleevi Quality Assurance Specialist
Michael Phillips Technical Director	Karen Kuoppala Operations Manager
Adam Alban EHS Coordinator	Chad Lancaster Waste Control Specialist
Brett VanDelinder Customer Service Manager	Pat McEntee Program Support Manager
Karen Kuoppala Operations Manager	William Rhodes VOA GC/MS Manager

Figure 4-1. Corporate and Laboratory Organization Charts

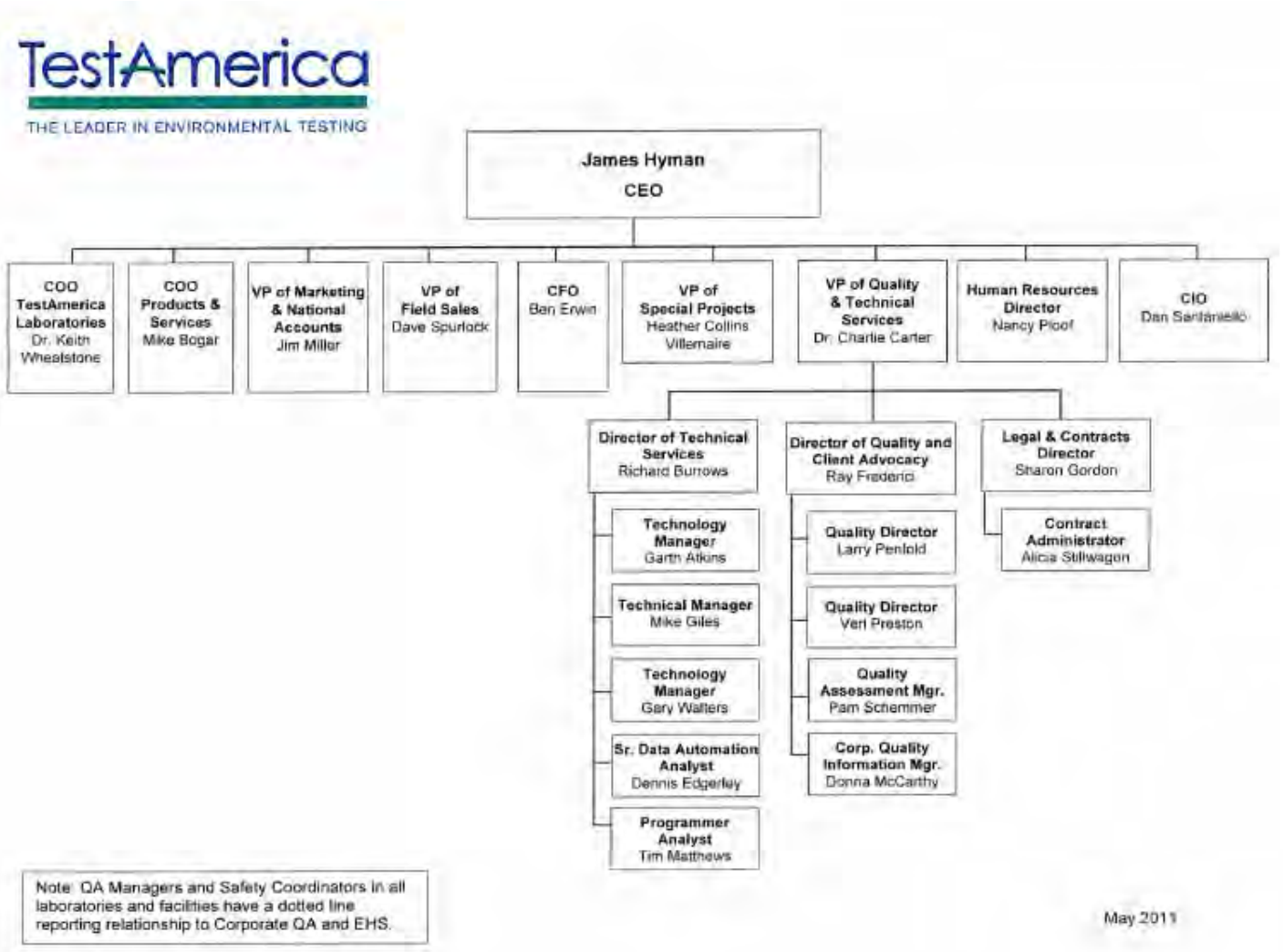
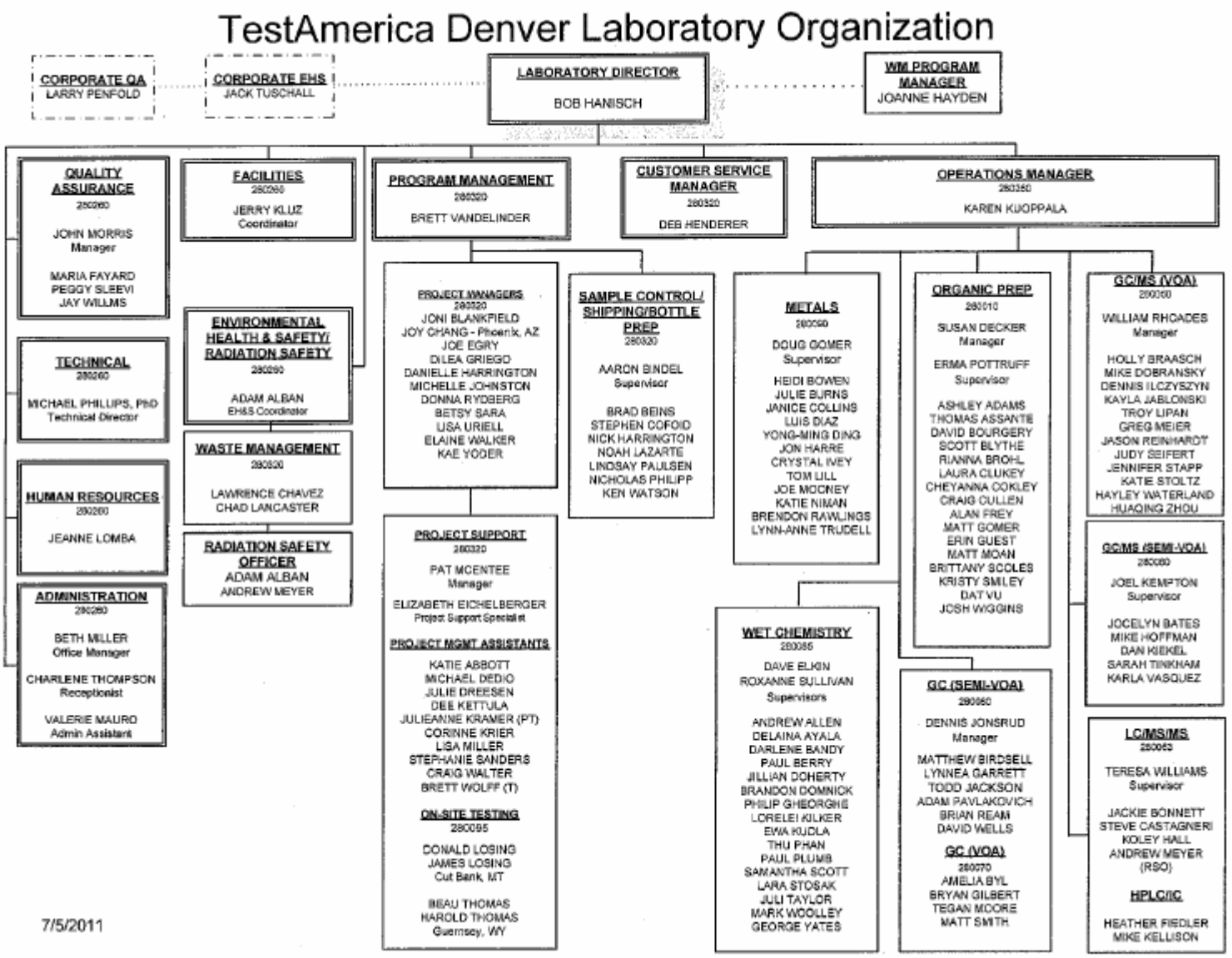


Figure 4-1. Corporate and Laboratory Organization Charts – con't



SECTION 5. QUALITY SYSTEM

5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.
- To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.
- Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002.)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

5.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit). Other terms may be used to reflect the method detection limit or reporting limit for specific programs.

5.5 Criteria for Quality Indicators

The laboratory maintains a *Quality Control Limit Summary* in the LIMS referred *to as "Method Limit Groups"* that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP DV-QA-003P, *Quality Assurance Program*.

5.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The control charting process is defined in detail in SOP DV-QA-003P. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Manager and QA Manager) and entered into the Laboratory Information Management System (LIMS). The LIMS maintains an archive of all limits used within the laboratory. Limits are entered into the Method Limit Groups according to the effective date. All historical limits can be pulled using the "Historical" feature in the LIMS. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

As the QC limits are calculated, QC charts are generated showing warning and control limits for the purpose of evaluating trends. Refer to SOP DV-QA-003P for a description of the control charting process and evaluation of trending.

5.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6. DOCUMENT CONTROL

6.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory specific SOP DV-QA-0010, *Document Control* provides additional information for the Denver laboratory procedures.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 Document Approval and Issue

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of

document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a department manager submits an electronic or hardcopy draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 Procedures for Document Control Policy

For changes to the QA Manual, refer to SOP No. DV-QA-001P, *Preparation and Management of Standard Operating Procedures (SOPs) and Other Controlled Documents*. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder G:\QA\READ\SOPS\ESOPS\ALL.

For changes to SOPs, refer to SOP No. DV-QA-001P, *Preparation and Management of Standard Operating Procedures (SOPs) and Other Controlled Documents.* The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions, white papers, protocols, and information are organized by department and document type in the QA office. Electronic versions are kept on the Public server in the QA folder under G:\QA\Edit\FORMS and G:\QA\READ\SOPS\Word Docs. The procedure for the care of these documents is in SOP DV-QA-001P.

6.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP DV-QA-0005, *Document Archiving Procedure.*

SECTION 7. SERVICE TO THE CLIENT

7.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

7.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Customer Service Manager (CSM) is considered adequate. The CSM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Project Manager
- Customer Service Representative
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts**.** TestAmerica Denver's Customer Service Department maintains copies of all signed contracts for reference locally.

7.3 Documentation

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Contracts filed by the CSM group are filed in locked fire proof cabinets.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory CSM and the Lab Director/Manager.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing. Unique or large programs generally have a Quality Assurance Summary prepared by the PM. This summary is posted on the public Outlook folders for anyone in the lab to access. The Quality Assurance Summary documents all requirements that are non-standard.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated in the Quality Assurance Summery and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 Client Communication

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors and/or Quality Assurance are available to discuss any technical questions or concerns that the client may have.

7.6 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 Client Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8. SUBCONTRACTING OF TESTS

8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required. Refer to SOP DV-QA-0027 for laboratory specific procedures.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies (e.g, USDA) or contracts (e.g, certain USACE projects)-may require notification prior to placing such work (see SOP DV-QA-0027 for laboratory specific procedures)**.**

8.2 Qualifying and Monitoring Subcontracators

Whenever a PM or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives, CSMs, or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the

use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers and Sales Personnel.

8.3 Oversight and Reporting

The PM or CSM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 Contingency Planning

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1.

Example: Verification of Subcontract Lab Status Form

TestAmerica Denver is responsible to our clients for on-going assurance that subcontracted analytical services meet TestAmerica Denver's expectations for quality. As part of this program, we require on-going verification that the following statements are true. Please return the completed form with the final report to TestAmerica Denver.

Laboratory Name: _____

	True	False	N/A	Comments
Your laboratory continues to hold current certifications as applicable to the requested fields of testing?				
Your laboratory has successfully completed PT samples for at least 2 of the last 3 of the requested fields of testing?				
Your laboratory has successfully completed method detection limits for the requested fields of testing within the last 12 months?				
There are no changes in equipment that affect the laboratory's capability to perform the requested fields of testing?				
There are no changes in qualified personnel that affect the laboratory's capability to perform the requested fields of testing?				
All testing is performed at the location to which the samples were delivered?				
Your laboratory does not have any OSHA, DOT, DoE, DoD, or EPA citations or pending investigations?				

Completed by: _____ on _____.
Name

SECTION 9. PURCHASING SERVICES AND SUPPLIES

9.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

9.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Material Request Sheet when requesting reagents, standards, or supplies. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Director), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Technical Director prior to placing the order. The purchasing manager or designee places the order.

9.3.2 Receiving

It is the responsibility of the shipping/receiving technician to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials where received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date cannot be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained within each department.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. To prevent a tank from going to dryness, close observation of the tank gauge must take place as pressure decreases towards 500 psig, or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical–Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in the LIMS. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager/Director, Operations Manager, and/or the Laboratory Director. If they agree with the

request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and the corporate office places the actual order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager / Director.

9.6 Suppliers

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technology Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10. COMPLAINTS

10.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following in SOP DV-QA-013P, *Customer Complaints.*

10.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP # DV-QA-013P.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

SECTION 11. CONTROL OF NON-CONFORMING WORK

11.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the

laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Refer to SOP# DV-QA-0031, *Nonconformance and Corrective Action System* for the procedure to handle such situations.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Director and QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run.

11.2 Responsibilities and Authorities

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance

Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

11.4 Prevention of NonConforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the

steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Operations Manager, Technical Manager/Director, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12. CORRECTIVE ACTION

12.1 Overview

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

12.2.1 Non-Conformance Report (NCM) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips.

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends
- Excessive revised reports
- Health and Safety violations

This will provide background documentation to enable root cause analysis and preventive action.

12.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

12.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

12.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM

Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

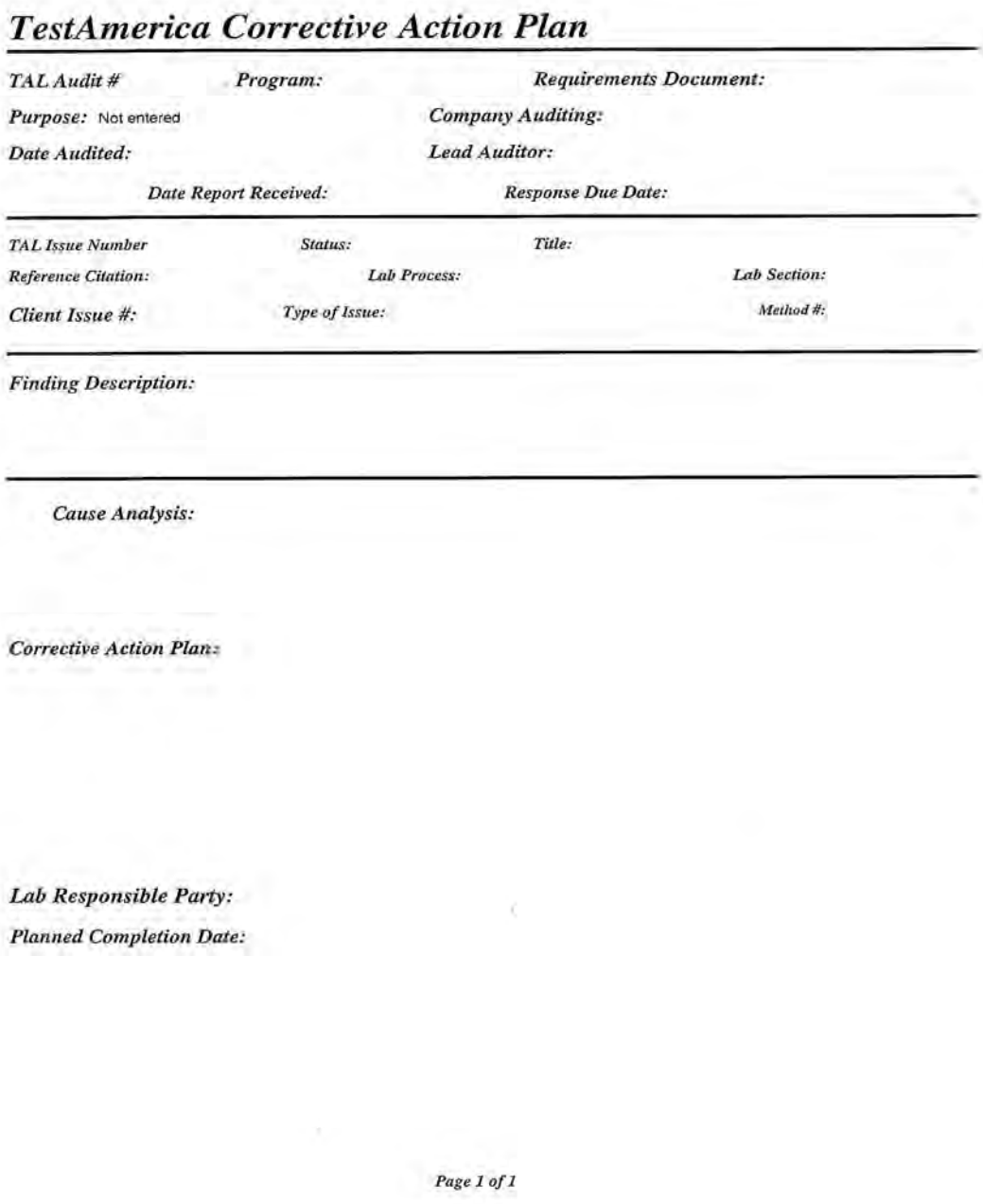
12.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example - Corrective Action Report



TestAmerica Corrective Action Plan

TAL Audit # *Program:* *Requirements Document:*

Purpose: Not entered *Company Auditing:*

Date Audited: *Lead Auditor:*

Date Report Received: *Response Due Date:*

TAL Issue Number *Status:* *Title:*

Reference Citation: *Lab Process:* *Lab Section:*

Client Issue #: *Type of Issue:* *Method #:*

Finding Description:

Cause Analysis:

Corrective Action Plan:

Lab Responsible Party:

Planned Completion Date:

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Table 12-1. Example – General Corrective Action Procedures

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank *(Analyst)*	- Instrument response < ½ RL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99. - Standard concentrations should bracket reporting limit. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits as defined in the method SOPs.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMS.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. See SOP DV-QA-003P for detailed corrective actions.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMS.	See SOP DV-QA-003P for detailed corrective actions.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	See SOP DV-QA-003P for detailed corrective actions.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	See SOP DV-QA-003P for detailed corrective actions.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc.	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 and DV-QA-019P.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated). See SOP DV-QA-013P.
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below ½ the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

SECTION 13. PREVENTIVE ACTION / IMPROVEMENT

13.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

13.2 Management of Change

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP # DV-QA-028P, *Management of Change.*

SECTION 14. CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

14.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic

or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the Department Manager or their designee.

Table 14-1. Record Index[1]

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.

[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or the Iron Mountain data storage facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained on-site at the laboratory for at least 3 months after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have longer retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 14-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

14.1.3 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information. In addition, refer to SOP # DV-QA-025P, *Electronic Data Backup.*

14.1.4 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set per SOP #DV-QA-0005, *Document Archiving Procedure*. Instrument data are stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data are lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP #DV-QA-0005, *Document Archiving Procedure*.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

14.2 Technical and Analytical Records

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

14.2.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and

19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

14.3 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a

description of the specific computational steps used to translate parametric observations into a reportable analytical value;

- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction,

validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS – no logbooks are used to record that data**.** Records are considered archived when noted as such in the records management system (a.k.a., document control.)

14.5.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

SECTION 15. AUDITS

15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and**,** when requested**,** to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing internal auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal

audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 50% of methods annually 100% of methods annually (DoD Labs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., MintMiner and Chrom AuditMiner) used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water (WS), Nonpotable Water (WP), Soil, Underground Storage Tank (UST).

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

15.3 Audit Findings

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16. MANAGEMENT REVIEWS

16.1 Quality Assurance Report

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Managers, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of

policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 Annual Management Review

The senior lab management team (Laboratory Director, Technical Managers, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel are to be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed).
- Compliance to the Ethics Policy and Data Integrity Plan, including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in a finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

SECTION 17. PERSONNEL

17.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular

area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience

Specialty	Education	Experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers– **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience
Technical Managers - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in the training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics is maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP DV-QA-0024.

17.4 Data Integrity and Ethics Training Program

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

18.1 Overview

The laboratory is a 54,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

18.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas.
- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

Refer to the following documents and procedures for specific requirements for microbiological laboratory facility requirements.

- Standard Methods, 20th Ed., 9020B, Sec. 2
- TNI V1M5, 1.7.3.7.a

18.4 Floor Plan

A floor plan can be found in Appendix 1.

18.5 Building Security

Building keys and alarm codes are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19. TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP DV-QA-001P.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*

- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC, DV-QA-0024) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months).

The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

19.4.3.1 Refer to SOP DV-QA-0024, Training.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the

precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with

acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte

initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. DV-QA-003P Determination of Method Detection Limits for Chemical Tests, for details on the laboratory's MDL process.

19.8 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3x the absolute value of the standard deviation.

If the IDL is > than the MDL, it may be used as the reported MDL.

19.9 Verification of Detection and Reporting Limits

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP DV-QA-003P, *Determination of Method Detection Limits (MDLs/DLs) for Chemical Tests* for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects,

TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

19.10 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

19.11 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.12 Estimation of Uncertainty of Measurement

19.12.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte.

The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/L, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/L, which could also be written as 1.0 +/- 0.5 mg/L.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats.

Note: Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.14 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP DV-QA-017P, *Electronic Reporting*. The laboratory is currently running the TestAmerica Laboratory Information Management System which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Sequel Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 **Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.14.1.2 **Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 **Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review

checklists, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*. and TestAmerica Denver SOP DV-QA-011P, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the batch folder and batch file in LIMS. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter (µg/L) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%**.** Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report. Refer to DV-QA-004P, *Rounding and Significant Figures* for details regarding the number of significant figures to report for each step in the process.

19.14.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. When this is not possible the data is entered into a logbook which becomes part of the raw data. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst reviews the values in LIMS against the raw results to check for errors. The instrument's calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the batch

file in LIMS. In addition, the data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are out lined in several SOPs (e.g. DV-QA-0003, *Sample Management and Chain of Custody*, DV-QA-0020, *Data Review*, and DV-QA-0022, *Data Package Assembly),* to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data DV-QA-011P, *Acceptable Manual Integration Practices*. The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.

19.14.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to

ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.14.4.7 A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a

poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. DV-QA-011P, entitled *Acceptable Manual Integration Practices.*

19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example - Demonstration of Capability Documentation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

4955 Yarrow Street
Arvada, CO 80002
303-763-0100

Analyst Name
June 5, 2009

DV-WC-0020
Method 300.0A and 9056
Analysis / Extraction

We the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.
3. A copy of the laboratory-specific SOP and applicable reference methods are available for all personnel on-site.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (*). These data are attached to this certification statement.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name Signature & Date

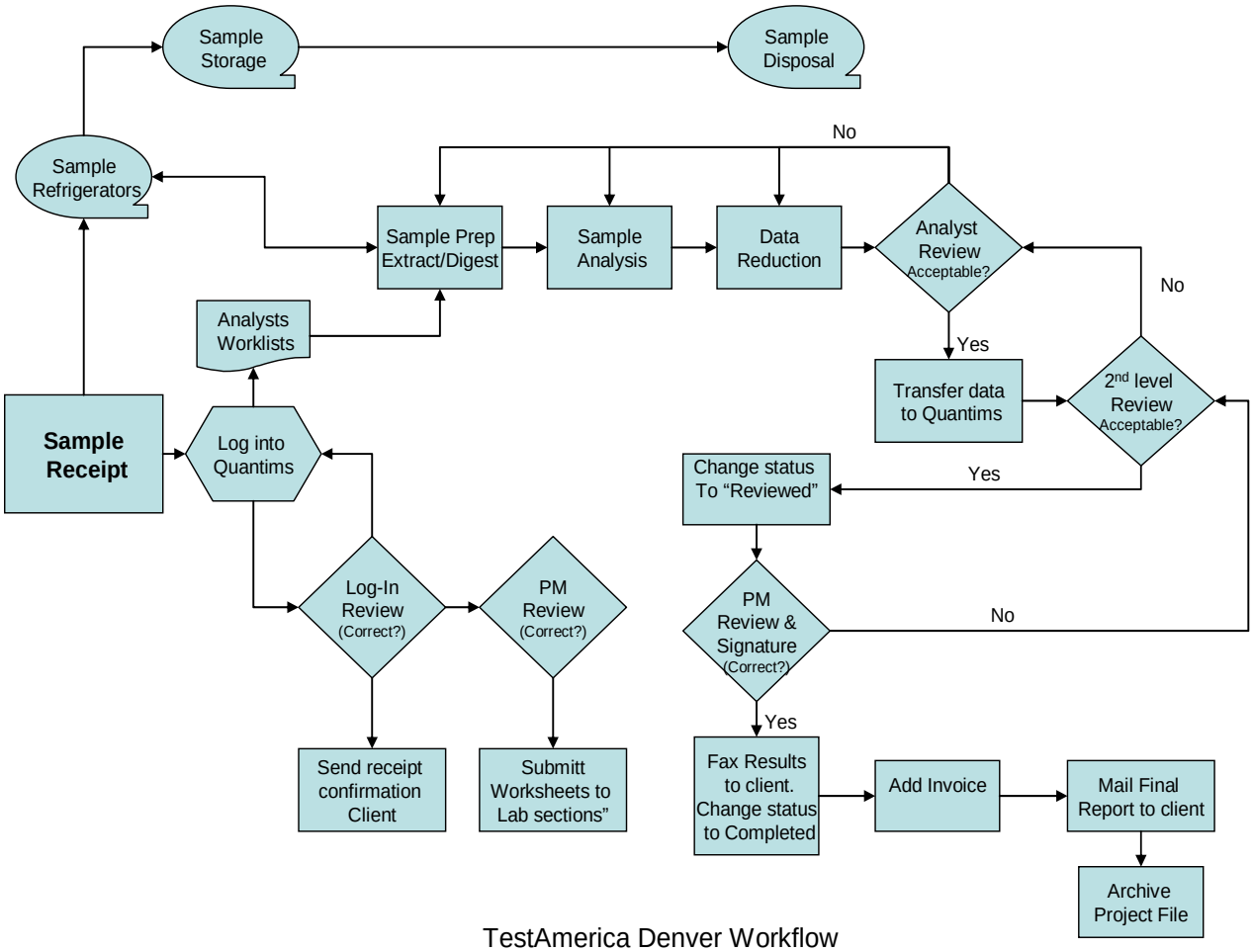
Operation Manager's Name Signature & Date

QA Manager's Name Signature & Date

* *True: Consistent with supporting data.*
Accurate: Base on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing
Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation

Revision 0 6/17/2009 G:\QA\Edit\FORMS\QA\IDOC Signature Page.doc

Figure 19-2. Example: Work Flow



TestAmerica Denver Workflow

SECTION 20. EQUIPMENT AND CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be/are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or

maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually

and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to SOP DV-QA-0014, *Balance Calibration Check*.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The mercury NIST thermometer is recalibrated every five (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in SOP DV-QA-0001, *Thermometer Calibration Procedure*.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored 7 days a week.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. Refer to an SOP DV-QA-0008, *Calibration and Verification of Mechanical Pipettes.*

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. Syringes are verified for syringe sizes of ≥ 100 µL are verified. Details are in DV-QA-0008.

20.3.6 Autoclaves

TestAmerica Denver uses an autoclave for sterilization of microbiological equipment and used media only. All information regarding the autoclave is maintained in the Autoclave, Coliform lot, and Monthly check logbook. The information recorded includes the date, contents, maximum temperature, total run time and the analyst's initials.

Demonstration of sterilization of the autoclave is performed each time of use with a Diack sterilization monitor, a maximum reading thermometer, and temperature sensitive tape. On a monthly basis, spore strips are used for the determination of effective sterilization.

The autoclaves timing device is checked on a monthly basis against a clock/watch and the actual time elapsed is documented.

Any maintenance that is performed on the autoclave (internally or by service contract) is recorded in the maintenance section of the check logbook.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as Disodium Iminodiacetate (IDA) analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GC/MS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument/method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a) When the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b) When the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the two conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be

reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Example: Instrumentation List

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
ICP	Thermo Fischer (025)	ICP 6500 S/N 20090307	2006	Yes	6010, 200.7
ICP	Thermo Fischer (026)	ICP 6500 S/N 20063207	2006	Yes	6010, 200.7
ICP/MS	Agilent ICP-MS (024)	7500 Series S/N JP51201530	2006	Yes	6020, 200.8
ICP/MS	Agilent ICP-MS (077)	7700 Series S/N JP09320047	2009	Yes	6020, 200.8
Mercury Analyzer	Cetac CVAA (033)	M-7500 S/N 08092QTA	2010	Yes	7470, 7471, 245.1
Mercury Analyzer	Cetac CVAA (034)	M-7500 S/N 0021105QTA	2010	Yes	7470, 7471, 245.1
Ion Chromatograph	Dionex (IC3)	DX-120 S/N 98040510	1997	Yes	300.0, 9056
Ion Chromatograph	Dionex (IC4)	AS 50 S/N 056537	2000	Yes	Hydrazine, MMH, UDMH
Ion Chromatograph	Dionex (IC5)	LC 20 S/N 0106180	2002	Yes	314.0
Ion Chromatograph	Dionex (IC6)	ICS 2000 S/N 03100162/03100118	2003	Yes	300.0, 9056, 9056A
Ion Chromatograph	Dionex (IC7)	ICS 2000 S/N 03100161/03100122	2003	Yes	300.0, 9056, 9056A
Ion Chromatograph	Dionex (IC8)	ICS 2000 RFIC S/N 08020954/08020762	2008	Yes	300.0, 9056, 9056A
Ion Chromatograph	Dionex (IC9)	ICS 3000 S/N 08020888	2008	Yes	Hydrazine, MMH, UDMH
TOC	LECO (LEC) S/N 3097	C632 (Solid) S/N 3097	2007	Yes	5310B, 9060
TOC	Shimadzu (SHI3)	TOC-V_{CPN} S/N H51404335027	2005	Yes	415.1, 9060, 5310B
TOC	Shimadzu (SHI2)	TOC-VCSH S/N 414445340	2004	Yes	415.1, 9060, 5310B
TOX	MCI (TOX10)	TOX-10 S/N 43F30338	1987	No	Not In Use
TOX	Thermo Euroglass (Thermo 3)	ECS 1200 S/N 993752/ 2008.283	2008	Yes	9020, 9021, 9023

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
TOX	Thermo Scientific (Thermo 2)	ECS 1200 S/N 993728/ 2004.119	2004	Yes	9020, 9021, 9023
UV/VIS	Thermo UV1	UV1 S/N 114403	2004	Yes	Not In Use
UV/VIS	Alpkem (Alp1)	A002393 S/N 908893427/ 906850834	1997	Yes	325.2, CN, Phenol
UV/VIS	Alpkem (Alp2)	A002393 S/N 917893398/ 912850458	1997	Yes	350.1, 353.2
UV/VIS	Konelab	Model 20 S/N P0518697	2003	Yes	365.1, 420.1, ASTM D516-02
UV/VIS	Astoria Pacific Analyzer	Astoria 2 S/N 200052	2005	Yes	351.2, 353.2, 365.1, 365.1 LL
UV/VIS	Alpkem (ALP3)	Flow Solution 3000 S/N 632-1010-49/60092	2010	Yes	350.1 (In Method Development)
Oil & Grease Extractor	Horizon Technology	3000 S/N: 03-1185	2004	No	1664A
Ion Analyzer	Orion Research	EA940 S/N PX94A	1985	No	Out of Service
Autotitrator (pH, Alkalinity, Conductance)	Man-Tech (AT2)	PC – Titrate PC-1000	2000	Yes	9040, 9045, 2320B, 2510B, 9050A
pH Meter	Thermo Orion / SensorLink	PerpHecTROSS Sure Flow pH/ ATC 93700 S/N: NQ211921	2003	No	9040, 9045, 4500-H B
pH Meter	Thermo Orion	SA 720 S/N: TVT71A	2005	No	1311
pH Meter	ThermoScientific	Orion 5 Star S/N: B27854	2009	No	2320B (Hand Titration), 2310B
Conductivity Meter	YSI	YSI 3200-115V S/N: 02G0765 AF	2009	No	Not In Use
Conductivity Meter	Orion	Orion 160 S/N: 42536014	1985	No	2510B, 9050A
Dissolved Oxygen Meter	YSI	5100 S/N 08D100984	2008	No	Not In Use
Dissolved Oxygen Meter		5100 S/N 02G0238	2008	No	405.1, 5210B

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
Turbidimeter		Micro 100	2001	No	180.1
Flashpoint	Herzog Pensky Martens	Model MP-329 S/N 043291648	2003	No	1010, 1010A
Spectro-photometer	HACH	DR/2010 S/N 990200012321	2007	No	410.4, 7196A, 3500 Fe D, 3500 Cr B, 3500 Cr D, 4500S^{-2} D, 420.1, 4500-NO_2 B
GC/MS Semivolatiles	Hewlett-Packard (B) S/N US00007283	6890 – GC 5973 – MSD	1999	Yes	8270, 625
GC/MS Semivolatiles	Hewlett-Packard (D) S/N US00007319	6890 – GC 5973 – MSD	1996	Yes	8270, 625
GC/MS Semivolatiles	Agilent Technologies (F) S/N US00036181	6890 – GC 5973N – MSD	1996	Yes	8270 SIM
GC/MS Semivolatiles	Agilent Technologies (K) S/N CN10332028	6890N – GC 5973 – MSD	2003	Yes	8270, 8270 SIM, 625
GC/MS Semivolatiles	Agilent Technologies (G6) S/N CN10933128	7890A – GC 5975C inert XL w/ triple Axis Detector	2010	Yes	8270
GC/MS Semivolatiles	Agilent Technologies (G4) S/N CN10438087	6890N – GC 5973 Inert – MSD	2004	Yes	8270 Best Practice
GC/MS Semivolatiles	Agilent Technologies (X4) S/N CN10438076	6890N – GC 5973 inert – MSD	2004	Yes	8270, 625
GC/MS Semivolatiles	Hewlett-Packard (Y) S/N US00007291	6890 – GC 5973 – MSD	1996	Yes	8270, 625
GC/MS Semivolatiles	Agilent Technologies (G5) S/N CN10605078	6890N – GC 5975 – MSD	2006	Yes	8270, 8270 SIM, 625
GC/MS Volatiles	Agilent Technologies (C) S/N US00007315 O·I Analytical S/N 14049	6890N – GC 5973 – MSD 4552 - Purge & Trap 4660 Concentrator	2002	Yes	8260

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
GC/MS Volatiles	Agilent Technologies (E) O·I Analytical S/N 13442	6890 – GC S/N-US00029543 5973 – MSD S/N –US93122910 4552 - Purge & Trap S/N - 13442 4660 – Concentrator S/N – G116466985P	2011	Yes	8260 SIM & 8260
GC/MS Volatiles	Hewlett-Packard (H) S/N 3336A60700 O·I Analytical S/N 14052	5890II – GC 5972 – MSD 4552 – Purge & Trap 4660 - Concentrator	1994	Yes	8260-Waters
GC/MS Volatiles	Hewlett Packard (P) S/N US00007321 O·I Analytical	6890 - GC 5973 – MSD 4552 – Purge & Trap 4660 - Concentrator	1999	Yes	8260
GC/MS Volatiles	Hewlett-Packard (G) S/N 3336A56276 Varian S/N 12751	5890 Series II - GC 5972 – MSD Archon Purge & Trap O·I 4560 - Concentrator	1996	Yes	8260
GC/MS Volatiles	Hewlett-Packard (J) S/N 3336A60701 Varian S/N 12726	5890II – GC 5972 – MSD Archon Purge & Trap O·I 4560 - Concentrator	1994	Yes	8260
GC/MS Volatiles	Agilent Technologies (R1) S/N LN10524033 O·I Analytical S/N 14043	6890N - GC 5973 Inert – MSD 4552 – Purge & Trap 4660 - Concentrator	1994	Yes	8260
GC/MS Volatiles	Hewlett-Packard (R2) S/N 336A53965 O·I Analytical S/N 14383	5890II - GC 5972 – MSD 4552 – Purge & Trap 4660 - Concentrator	1995	Yes	8260

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
GC/MS Volatiles	Hewlett-Packard (Z) O·I Analytical	5890II – GC 5972 – MSD S/N 3336A60013 Archon Purge & Trap O·I 4660 – Concentrator S/N G109466777	1996	Yes	8260
GC/MS Volatiles	Agilent Technologies (GC/MS1) O·I Analytical	6890N – GC 5973 – MSD S/N CN10420009 4552 – Purge & Trap 4660 - Concentrator S/N 14593	2004	Yes	8260
GC/MS Volatiles	Agilent Technologies (G2) Varian	6890N – GC 5973 – MSD S/N CN10421078 Archon Purge & Trap O·I 4660 - Concentrator S/N MS0902W012	2004	Yes	8260
GC/MS Volatiles	Hewlett-Packard (Q) Varian	6890 – GC 5973 – MSD S/N US0000021949 Archon Purge & Trap O·I 4560 - Concentrator S/N 12750	2001	Yes	8260
GC/MS Volatiles	Hewlett-Packard (I) S/N 2643A11361 Tekmar Dohrmann Headspace Autosampler S/N US03038002	5890 – GC 7000	2003	Yes	Volatile Screening
GC/MS Volatiles	Hewlett-Packard (T) S/N 2750A14928 Tekmar Dohrmann Headspace Autosampler S/N US01198005	5890 Series II – GC 7000HT	2001	Yes	Volatile Screening
GC Semivolatiles	Hewlett-Packard (A) S/N 2750A16891	5890 Dual FID	1987	Yes	8015 Alcohol

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
GC Semivolatiles	Hewlett-Packard (C) S/N US00029514	6890 Dual ECD	1999	Yes	608, 8081
GC Semivolatiles	Hewlett-Packard (D) S/N DE00020818	6890 Dual NPD	1997	Yes	614, 8141
GC Semivolatiles	Agilent Technologies (D2) S/N US10521035	6890N Dual NPD	2004	Yes	614, 8141
GC Semivolatiles	Hewlett-Packard (E) S/N 3121A35858	5890II Dual ECD	1992	Yes	504.1, 8011
GC Semivolatiles	Hewlett-Packard (M) S/N US00024143	6890 Dual ECD	1999	Yes	615, 8151
GC Semivolatiles	Agilent Technologies (P1) S/N US10418019	6890N Dual ECD	2004	Yes	608, 8081
GC Semivolatiles	Agilent Technologies (P2) S/N US10418024	6890N Dual ECD	2004	Yes	608, 8081
GC Semivolatiles	Agilent Technologies (P3) S/N US10418023	6890N Dual ECD	2004	Yes	608, 8082
GC Semivolatiles	Hewlett-Packard (R) S/N 3336A55030	5890II Dual ECD	1994	Yes	Not is use
GC Semivolatiles	Hewlett-Packard (T) S/N 2536A05971	5890 Dual NPD	1999	Yes	607, 8070
GC Semivolatiles	Hewlett-Packard (U) S/N US00063217	5890II Single FID	1999	Yes	8015 DRO
GC Semivolatiles	Hewlett-Packard (V) S/N 2631A08686	5890 Dual ECD	1990	Yes	8081 (limited use)
GC Semivolatiles	Hewlett-Packard (W) S/N 3126A36250	5890II Dual ECD	1990	Yes	608, 8082
GC Semivolatiles	Hewlett-Packard (Z2) S/N 3336A51924	5890II Dual FID	1990	Yes	8015 DRO
GC Semivolatiles	Agilent (U2) S/N CN10942072	7890A Dual FID	11/11/09	Yes	8015 DRO

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
GC Volatiles	Hewlett-Packard (B) S/N 3019A28634 Tekmar	5890 Series II Dual PID / FID LSC 2000 Concentrator S/N 90142014 ALS 2016 Purge & Trap S/N 89108007	1990	Yes	8021 GRO
GC Volatiles	Hewlett-Packard (F) Tekmar	5890II Dual ELCD LSC 2000 Concentrator S/N 88305008 ALS 2016 Purge & Trap S/N 90129029	1990	Yes	Retired/ Parts only
GC Volatiles	Hewlett-Packard (J) S/N US00026194 Tekmar Dohrmann US02296004	6890 Dual FID HS Autosampler 7000 HT	1997	Yes	RSK-175
GC Volatiles	Hewlett-Packard (K) S/N 2843A19497 Tekmar	5890A Dual PID Single FID LSC 2000 Concentrator S/N 92098003 ALS 2016 Purge & Trap S/N 92101007	1988	Yes	8015, 8021 Aromatics, 8021 GRO
GC Volatiles	Hewlett-Packard (L) S/N 2336A00164 Tekmar	5890A FID LSC 2000 Concentrator S/N 89283001 ALS 2016 Purge & Trap S/N 90121028 ALS 2032 Purge & Trap S/N 94300004	1988	Yes	8015 GRO
GC Volatiles	Hewlett-Packard (P) S/N 2518A05337	5890A Dual PID Single FID LSC 2000	1990	Yes	Currently Out of Service

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
	Tekmar	Concentrator S/N 89310005 ALS 2016 Purge & Trap S/N 90100036			
GC Volatiles	Agilent Technologies (S-1) S/N US10341120 O I Analytical S/N 14046	6890 Dual PID/ Dual ELCD 4552 – Purge & Trap 4660 - Concentrator	2003	Yes	8021
GC Volatiles	Hewlett-Packard (Y) S/N 2843A19484 Tekmar	5890A PID/FID LSC 3000 Concentrator S/N 93132006 ALS 2016 Purge & Trap S/N 91112002 ALS 2032 Purge & Trap S/N 88145006	1988	Yes	Screen only
GC Volatiles	Hewlett-Packard (H) Tekmar	5890A Dual PID Single FID LSC 2000 Concentrator S/N 90100002 ALS 2016 Purge & Trap S/N 88145007	1988	Yes	8015, 8021 Aromatics, 8021 GRO
HPLC	Hewlett-Packard (G) S/N DE91609974	1100 Multiple wavelength UV/ Fluorescence detectors	1999	Yes	8310
HPLC	Agilent Technologies (G2) S/N US83102106 (Binary Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2011	Yes	8310, 8330
HPLC	Agilent Technologies (Q) S/N DE11120993 (Quat Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2001	Yes	8330

Instrument Type	Manufacturer/Lab Name	Model/Serial Number	Purchase Date	Auto-sampler	Method Performed
HPLC	Agilent Technologies (X3) S/N DE33236507 (Quat Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2004	Yes	8330
HPLC	Agilent Technologies (X4) S/N DE22601691 (Quat Pump)	1100 Multiple wavelength UV/ Fluorescence detectors	2010	Yes	8330
HPLC/MS/MS	Micromass/Waters 2790 HPLC Inlet S/N VB118 (LCMS1) plus Dionex AS50 Autosampler, LC30 Chromatography Oven, CD25 Conductivity Detector	Quattro Ultima	2000	Yes	8321, 6860
HPLC/MS/MS	Micromass/Waters Acquity UPLC Inlet (LCMS3) S/N VAB661	Quattro Premier XE	2004	Yes	8321
HPLC/MS/MS	Micromass/Shimadzu 10 Avp HPLC Inlet (LCMS2) plus Shimadzo Inlet SIL-10AD, Shimadzo UV-VIS Detector SPD-10A, Dionex Ion Chromatography ICS 2000 S/N VB304	Quattro Ultima	2001	Yes	8321
HPLC/MS/MS	Micromass/Waters 2695 HPLC Inlet (LCMS4) S/N QAA632	Quattro Micro AP1	2006	Yes	8321
HPLC/MS/MS	Agilent Technologies S/N US95270371	6400 Triple Quad LC/MS	2010	Yes	8321
CI/MS/MS	Varian (CIMS1) S/N 1200-680	1200L MS/MS CP-3800 GC	2004	Yes	Low Level NDMA

Support Equipment				
Instrument Type	**Manufacturer**	**Model**	**Quantity**	**Location**
Centrifuge	Sorvall Legend T	Sorvall Legend T	1	Metals
Hot Block	Environmental Express	SC100	11	Metals
Sonic Bath	Bransonic	Bransonic	1	Metals
Hot Block	Thermo Scientific Precision	Thermo Scientific Precision	4	Metals
Incubator	Fisher Scientific	Incubator	1	Wet Chemistry
Water Bath	Fisher Scientific	Isotemp 2150	1	Wet Chemistry
Autoclave	Tuttnaver	2340M	1	Wet Chemistry
Magnifier	Darkfield Quebec	Colony Counter	1	Wet Chemistry
Incubator	Precision Scientific	Spore Strip Incubator	1	Wet Chemistry
Incubator	Fisher Scientific	Low Temperature Incubator	1	Wet Chemistry
Incubator	Thermo Electron Corporation	Thermo Electron Corporation	1	Wet Chemistry
TOX Sample Preparation	Microcoulometric Titration System	Microcoulometric Titration System	5	Wet Chemistry
Cyanide Digestor	Westco Scientific Instruments, Inc.	Westco Scientific Instruments, Inc.	1	Wet Chemistry
Centrifuge	Beckman	Beckman G- D-G	1	Wet Chemistry
COD Digestor	HACH	DRB 200	2	Wet Chemistry
Digestion System w/ Controller	A I Scientific	AIM 600/AIM 500	1	Wet Chemistry
Solvent Evaporator w/Digital Temperature Control System	UA-SYS	UA-SYS Heating System S-EVAP KD	1	Wet Chemistry
Oil & grease Machine w/ SPE-DEX 3000 Controller/ Speed VAP II 9000 Solvent	Horizon Technology	3000 XL	1	Wet Chemistry

Support Equipment				
Instrument Type	**Manufacturer**	**Model**	**Quantity**	**Location**
Evaporation System VAC Generator				
Cool Flow 25 NES Lab Kontes w/Midi Vap 2000	Scientific Glassware Instruments	Scientific Glassware Instruments	1	Wet Chemistry
Oven (D)	VWR	1370 GD	1	Wet Chemistry
Oven (C)	VWR	1370 G	1	Wet Chemistry
Oven (B)	VWR	1370 FM	1	Wet Chemistry
Oven (E)	Fisher Scientific	Fisher Scientific	1	Wet Chemistry
Oven (A)	Yamato	Mechanical Convection Oven DKN 810	1	Wet Chemistry
Centrifuge	IEC Clinical	IEC Clinical	1	Mass Spectrometry
Sonicator Bath	Branson	5510	2	Explosive Prep
Water Bath	Waterlow	Waterlow	2	North Prep
Oven	Fisher Scientific	Fisher Scientific	1	North Prep
Turbo Vap	Caliper Life Science	Turbo Vap II	2	North Prep
Seperatory Funnel Rotators	Ap & R Machine Tool	Ap & R Machine Tool	5	North Prep/South Prep
Lab Ultra	ELGA	Pure lab Ultra	1	North Prep
Microwave Extraction	CEM Corporation	MARSXpress Xtraction	1	North Prep
Sonicator	Fisher Scientific	550 Sonic Dismembrator	2	North Prep
Sonicator	Heat Systems	Sonicator Ultrasonic Processor XΔ	1	North Prep
Sonicator	Misonix	Sonicator 3000	2	North Prep
Sonicator	Heat Systems	W-385	2	North Prep

Support Equipment				
Instrument Type	**Manufacturer**	**Model**	**Quantity**	**Location**
Water Bath	Waterlow	Waterlow	2	North Prep
Drying Oven	Blue M	Temp-O-Loy Amecling Oven	1	North Prep
Turbo Vap (A)	Zymark	Turbo Vap II	1	North Prep
N-Evap	Organomation Associates, Inc.	N-Evap II Nitrogen Evaporator	2	North Prep
Centrifuge	International Equipment Company	Model K	1	North Prep
Vacuum Manifold	Waters	Vacuum Manifold	2	South Prep
N-Evap	Organomation Associates, Inc.	N-Evap II Nitrogen Evaporator	1	South Prep
Shaker	New Brunswick Scientific	Innova 2100	1	South Prep
Muffle Furnance	Lindberg	Lindberg	1	South Prep
Sonicator	Branson	Branson 2210	1	South Prep
Centrifuge	International Equipment Company	Model K	1	TCLP
Rotary Agitation Apparatus			3	TCLP Prep
Oven	Labline	L-C Oven	1	TCLP
Oven	Fischer Scientific	Isotemp	1	TCLP
Oven	Precision	Oven	1	TCLP
Balance	Denver Instruments	Model: TP1502 S/N:24750526	1	GC SVOA Hood #31
Balance	Denver Instruments	Model: TP 1502 S/N: 24750525	1	GC SVOA Hood #32
Balance	Mettler	Model: AE 160 S/N: C33519	1	Metals - ICPMS
Balance	Mettler	Model: PM4000 S/N: M28318	1	Metals Prep
Balance	Denver Instruments	Model: TP 3102 S/N: 24750837	1	Hg Prep Room
Balance	Mettler	Model: AE 260 S/N: H52017	1	MS VOA Standards Prep Room

Support Equipment				
Instrument Type	**Manufacturer**	**Model**	**Quantity**	**Location**
Balance	Denver Instruments	Model: TP 3102 S/N: 24950431	1	TCLP room
Balance	Denver Instruments	Model: TP 323 S/N: 24850252	1	MS VOA Hood #36
Balance	Denver Instruments	Model: TP 323 S/N: 24650437	1	MS VOA Hood #37
Balance	Mettler	Model: AE 240 S/N: I23294	1	Stnds & Aliquotting Hood #58
Balance	Denver Instruments	Model: TP 3102 S/N: 24750836	1	North Prep - MIS room
Balance	Denver Instruments	Model: TP 6101 S/N: 24750402	1	North Prep
Balance	Denver Instruments	Model: TP 6101 S/N: 24750396	1	North Prep
Balance	Denver Instruments	Model: TP 214 S/N: 24450835	1	North Prep
Balance	Denver Instruments	Model: TP 6101 S/N: 24350888	1	North Prep
Balance	Denver Instruments	Model: TP 214 S/N: 24850570	1	Wet Chem
Balance	Denver Instruments	Model: TP 6101 S/N: 24750399	1	Wet Chem
Balance	Denver Instruments	Model: TP 6101 S/N: 24950441	1	Wet Chem
Balance	Denver Instruments	Model: TP 1502 S/N: 24650239	1	Wet Chem
Balance	Denver Instruments	Model: TP 3102 S/N: 24950432	1	Wet Chem
Balance	Mettler	Model: AE 163 S/N: D91301	1	Wet Chem /South Prep
Balance	Mettler	Model: PM 4600 S/N: H31422	1	Wet Chem
Balance	Satorius	Model: CP124S S/N: 19350788	1	Wet Chem Soil TOC
Balance	Denver Instruments	Model: TP 214 S/N: P214088008	1	South Prep

Table 20-2. Example: Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Cetak and Perkin Elmer Mercury Analyzers	• Check silica gel in drying tube • Change Lamp • Clean cell and aspirator in aqua regia • Check pump tubing and pump flow • Check Waste Container • Fill reductant bottle with 10% Stannous Chloride and check acid reagent	As needed As needed Monthly Daily Daily Daily
ICP	• Check pump tubing • Fill Argon humidifier with water • Check fluid level in waste container • Clean or replace air filters • Check torch for residue • Check nebulizer flow • Clean nebulizer and drain chamber • Fill rinse solution/ IS solution • Replace capillary tubing/sipper probe • Check internal fluid reservoir • Change internal cooling fluid	Daily Weekly Daily As needed Daily Daily As needed Daily As needed Monthly Yearly
ICP MS	• Change pump tubing • Check level of tuning solution • Check waste container • Load printer with paper • Check air filters • Replace coolant on chiller • Clean or change nebulizer • Clean or replace torch • Replace capillary tubing • Change oil in vacuum pumps • Remove and clean cones	Daily Daily Daily Daily Monthly Bi-annually As needed As needed As needed As needed As needed
UV-Vis Spectrophotometer	• Clean ambient flow cell • Precision check/alignment of flow cell • Wavelength verification check	As required As required Semi-annually
Colorimetric Analyzer	• Clean detector • Clean filters • Check tubing • Clean sample probe shaft • Clean pump, diluter, and XYZ sampler. • Lubricate pump roller	Daily Daily Daily Daily Monthly Semi-annually

Instrument	Procedure	Frequency
Ion Chromatograph	• Check plumbing for leaks • Check gases • Check pump pressure • Check eluent level • Check conductivity meter • De-gas pump head when flow is erratic • Change analytical columns and bed supports guard • Check and replace any damaged/discolored tubing • Clean conductivity cell • Lubricate left hand position	Daily Daily Daily Daily Daily As needed As needed As needed As needed As needed
Total Organic Halide Analyzer	• Check electrodes/polish if needed • Replace dehydrating fluid /electrolyte fluid • Clean quartz boat • Perform cell performance check • At the end of each day of use, wash out the absorption module, empty the electrolyte and fill chamber with DI water, empty dehydrator tube • Clean or replace pyrolysis tube • Clean titration cell • Replace reference electrode fluid • Change quartz wool • Replace o-rings and seals	Daily Daily Daily Daily Daily As needed As needed As needed As needed As needed
Hewlett Packard GC/MS	• Check inlet pressure • Check temperature of inlet, detector, verify temperature program • Check Septa and clean injection port • Check carrier gas supply • Check tune parameters • Check oil levels in mechanical pumps and the diffusion pump if the vacuum is insufficient • Replace electron multiplier • Clean Source • Replace filaments • Change rough pump oil and exhaust filters • Relubricate the turbomolecular pump-bearing wick	Daily Daily Daily Daily Daily As needed As needed As needed As needed As needed Annually Annually
Gas Chromatograph	• Check carrier gas supply • Check temperatures of inlet, detectors, verify temperature program • Check septa clean injection port or replace injection port liner and cut column if needed • Reactivate carrier gas drying agents • Replace or repair flow controllers if constant flow cannot be maintained	Daily Daily As needed As needed As needed

Instrument	Procedure	Frequency
Electron Capture Detector (ECD)	• Detector wipe test (Ni-63) • Detector cleaning	Semi-annually As needed
Flame Ionization Detector (FID)	• Detector cleaning	As needed
Nitrogen Phosphorus Detector (NPD)	• Replace bead • Replace ceramic rings	As needed As needed
Photoionization Detector (PID)	• Change O-rings • Clean lamp window	As needed As needed
HPLC	• Check level of eluent vessels • Check gas supply • Change pump seals • Change the column frit • Change fuses in power supply • Filter all samples • Change autosampler rotor or oil autosampler slides • Change or backflush columns	Daily Daily Semi-annually or as required As needed As needed Daily As needed As needed
APCI/ESI LC/MS/MS	• Check solvent reservoirs • Verify that pump is primed and operating pulse free • Verify temperatures for capillary heater/vaporizer heater • Verify pressure of manifold/fore-pump • Verify that corona and multiplier are functional • Clean Lenses • Clean skimmer • Replace column • Oil autosampler • Change autosampler filters • Replace sample inlet tube • Replace fused silica tubing at ESI interface • Replace rough pump oil • Replace turbo pump oil • Vacuum system components including fans and fan covers	Daily Daily Daily Daily Daily As needed As needed As needed As needed As needed As needed As needed As needed Semi-annually Annually Annually
Balances	• Class “S” traceable weight check • Clean pan and check if level • Field service	Daily, when used Daily At least Annually
Sonicator	• Inspect probe for etching/pitting • Tune sonicator assembly • Disassemble and clean probe tips	Daily Weekly As needed
Conductivity Meter	• Standardize with KCL • Conductivity cell cleaning • Check probes and cables	Daily As needed As needed

Instrument	Procedure	Frequency
Flash Point Tester	• Check stirrer • Check tubing • Check gas supply • Check thermometer against NIST thermometer	Daily Daily Daily Daily, when used
Digestion Block	• Check with NIST thermometer	Annually
Turbidimeter	• Check light bulb • Inspect cells • Clean housing	Daily, when used Monthly Monthly
Deionized/Distilled Water	• Conductivity check • System cleaning • Replace cartridge & large mixed bed resins	Daily As needed As needed
Drying Ovens	• Temperature monitoring • Temperature adjustments	Daily As required
Refrigerators/ Freezers	• Temperature monitoring • Temperature adjustment • Defrosting/cleaning	Daily As required As required
pH/Specific Ion Meter	• Calibration/check slope • Clean electrode	Daily As required
BOD Incubator	• Temperature monitoring • Coil and incubator cleaning	Daily Monthly
Centrifuge	• Check brushes and bearings	Every 6 months or as needed
Water baths	• Temperature monitoring • Water replaced	Daily Monthly or as needed

SECTION 21. MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

Calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty.

The uncertainty calculations of the calibration laboratory are supported by uncertainty budgets and are represented by expanded uncertainties typically using a coverage factor of k=2 to approximate the 95% confidence level. This explanation accompanies the measurement result and the associated uncertainty.

The tolerance uncertainty ratio (TUR) is calculated using the expanded uncertainty of the measurement, not the collective uncertainty of the measurement standards. A statement to this effect accompanies the TUR along with the coverage factor and confidence level.

The calibration report or certificate submitted to TestAmerica Denver contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis

upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

The calibration laboratory supports in-house calibration systems: documented procedures for in-house calibrations, evidence by a report, certificate, or sticker, for an appropriate amount of time; training records of calibration personnel; certificates from accreditation services demonstrating traceability to national or international standards of measurement; procedures for evaluating measurement uncertainty; timely and documented recalibration of reference standards. When subcontracting to a calibration laboratory, TestAmerica Denver does not use a firm who subcontracts the work.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation) with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the ‘true’ value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as Disodium Iminodiacetate (IDA) analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), *Solvent and Acid Lot Testing and Approval*.]

All manufacturer or vendor supplied Certificates of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in the analytical groups and by QA on the public drive. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to Denver SOP DV-QA-0015, *Verification and Storage of Calibration Standards.*

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date

- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID from LIMS
- Special Health/Safety warnings if applicable

Records must be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the LIMS and TestAmerica Intranet Oasis.

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

SECTION 22. SAMPLING

22.1 Overview

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However, there are some programs that determine holding time

compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located in SOP # DV-QA-0023, *Subsampling.*

SECTION 23. HANDLING OF SAMPLES

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time

- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

23.1.2 Legal / Evidentiary Chain-of-Custody

All samples are tracked through the LIMS software program to ensure internal chain of custody and cradle to grave tracking of each sample container. If samples are identified for legal/evidentiary purposes on the COC an internal COC can be generated from the LIMS and included in the data package.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

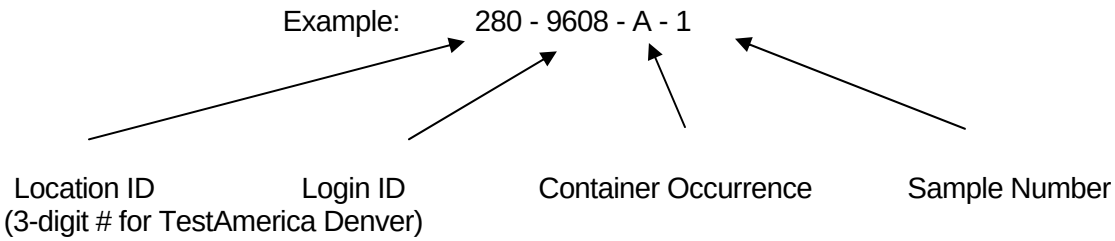
23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Condition Upon Receipt Anomaly Form (CUR) and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record. Refer to SOP DV-QA-0003, *Sample Management and Chain of Custody* for detailed information on receipt of samples.

23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Denver Laboratory (Location 280). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 280 - 9608 - A - 1 - **A** ←——— **Secondary Container Occurrence**

Example: 280-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1st occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Note: North Carolina requires that they be notified when samples are processed that do not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. DV-QA-0003, *Sample Management and Chain of Custody.*

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix, except metals sample containers which may be stored unrefrigerated*.* In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator, document the transfer of containers in LIMS and place them on carts, analyze the sample, and return the remaining sample to the refrigerator from which it originally came, documenting the return in LIMS. Empty containers are stored in the sample archive area until disposal, this transfer is documented in LIMS. All samples are kept in the refrigerators until the project is invoiced. At this time, the samples will be retained for an additional thirty days, either in the refrigerators, or in the sample archive area. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues. Upon disposal, the drum number used for disposal is logged into LIMS.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in a designated area. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, the analyst will notify login staff so the hazardous sample is properly labeled as such. The sample itself is clearly marked with a label reading “HAZARDOUS”, “PCBs” or “FOREIGN SOIL”. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility; refer to SOP DV-QA-0019, *Quarantine Soils Procedure* for more detail.

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: DV-HS-0005, *Excess Sample Material Management)*. All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

All documentation and correspondence concerning the disposal of samples is kept on file. The LIMS Internal Chain of Custody software allows tracking for each sample container from the time of sample receipt through the disposal process, including such detail as the identifying number of the waste drum used for disposal. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Hazardous Waste Manifest will be prepared to document the disposal of each drum. Additional detail is in SOP DV-HS-0004, *Hazardous Waste Manifesting*.

Figure 23-1. Example: Chain of Custody (COC)

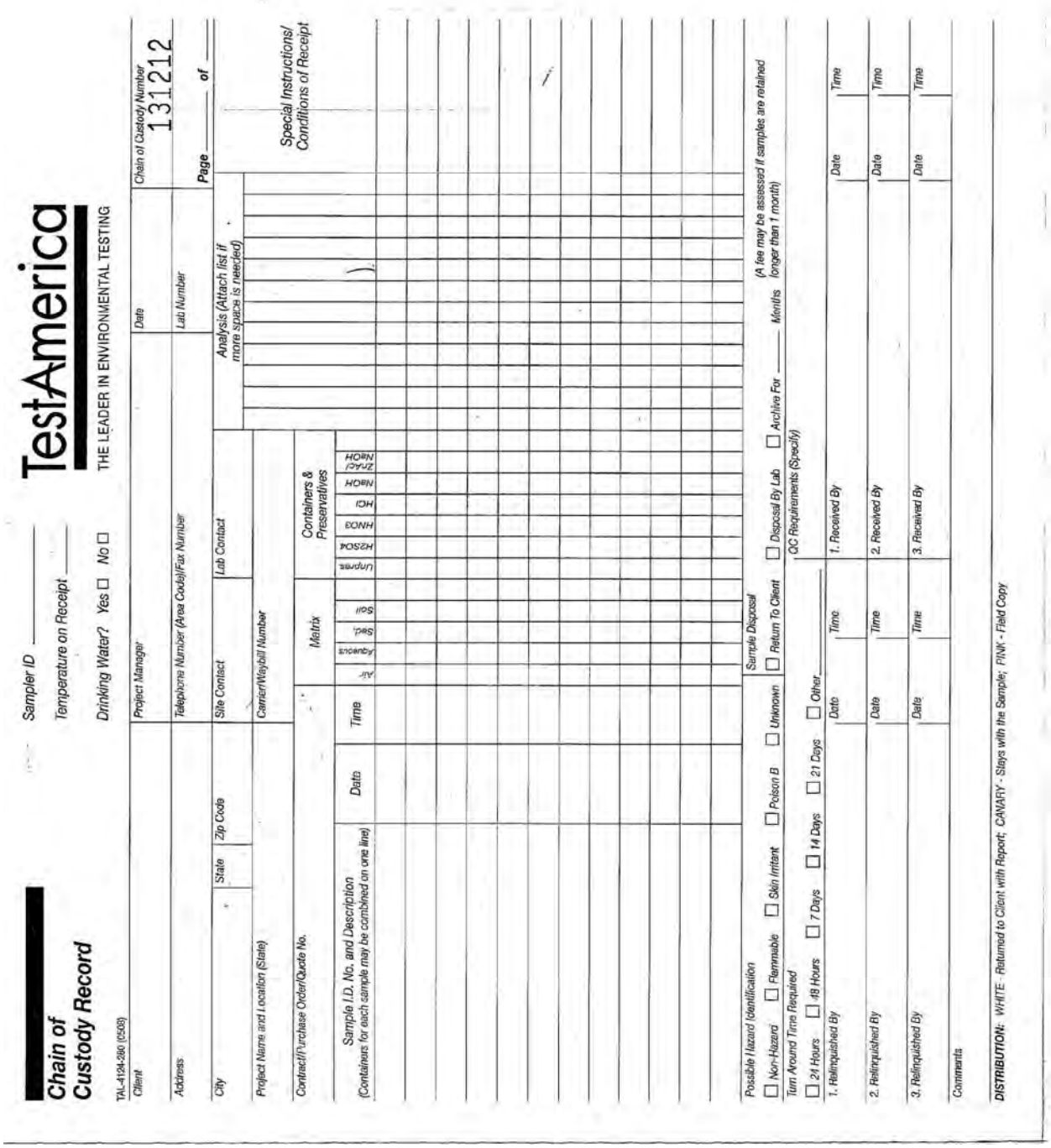


Figure 23-2. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - Client name, address, phone number and fax number (if available)
   - Project name and/or number
   - The sample identification
   - Date, time and location of sampling
   - The collectors name
   - The matrix description
   - The container description
   - The total number of each type of container
   - Preservatives used
   - Analysis requested
   - Requested turnaround time (TAT)
   - Any special instructions
   - Purchase Order number or billing information (e.g. quote number) if available
   - The date and time that each person received or relinquished the sample(s), including their signed name.
   - The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
   - Information must be legible

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide**.**

5) Most analytical methods require chilling samples to 4^{o} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{o} C and above freezing (0^{o}C). For methods with other temperature criteria (e.g. some bacteriological methods

require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2^{o} C of the required temperature or within the method specified range.

Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.

- **FOR WATER SAMPLES TESTED FOR CYANIDE (by Standard Methods or EPA 335)**

  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.

  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

  - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

6) Sample Holding Times
   - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.

   - Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field

sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

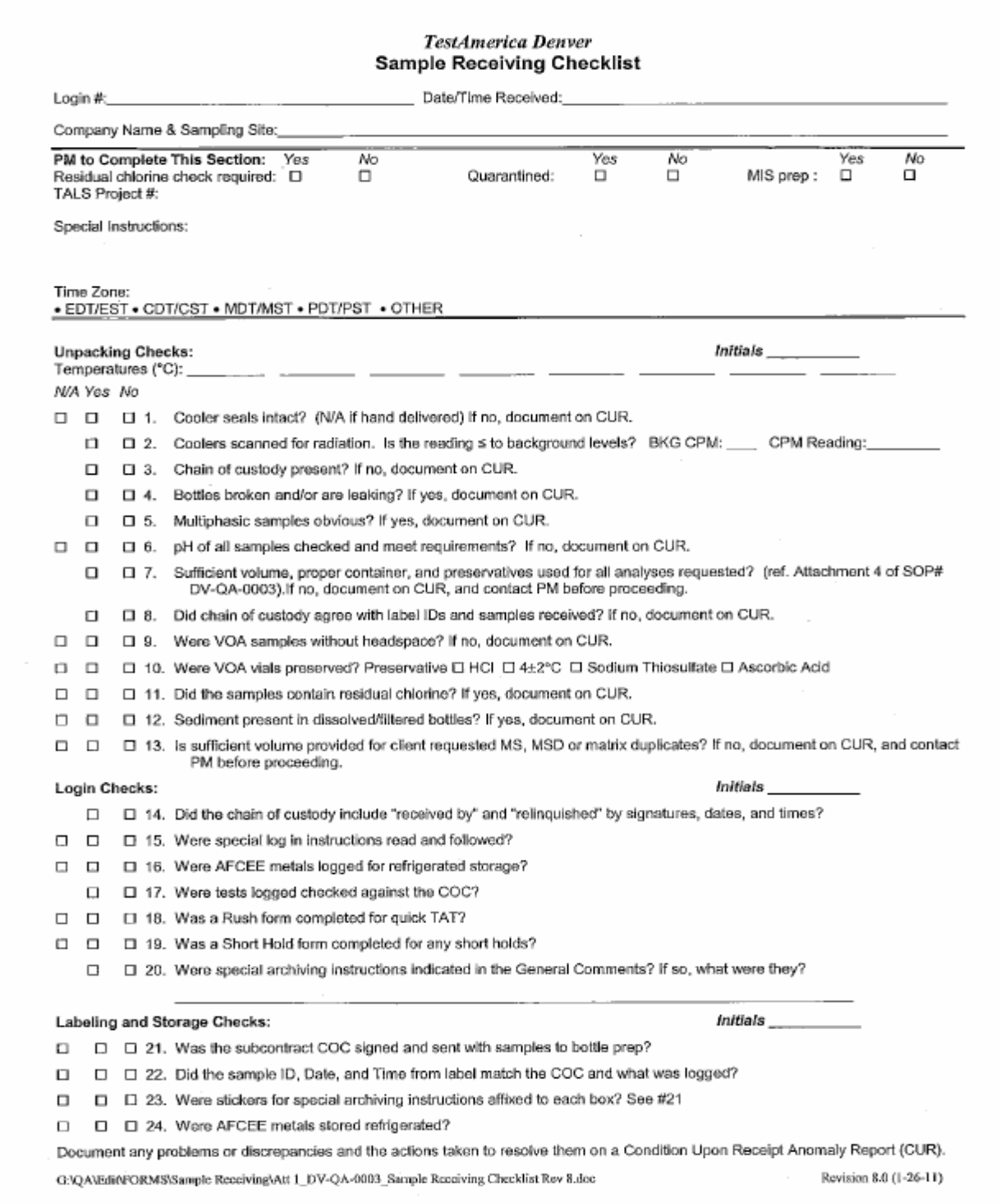
7) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

8) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

9) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

Figure 23-3. Example: Cooler Receipt Form



TestAmerica Denver
Sample Receiving Checklist

Login #:_____ Date/Time Received:_____

Company Name & Sampling Site:_____

PM to Complete This Section:	Yes	No		Yes	No		Yes	No
Residual chlorine check required:	☐	☐	Quarantined:	☐	☐	MIS prep :	☐	☐

TALS Project #:

Special Instructions:

Time Zone:
• EDT/EST • CDT/CST • MDT/MST • PDT/PST • OTHER

Unpacking Checks: ***Initials*** _____

Temperatures (°C): _____ _____ _____ _____ _____ _____ _____ _____

N/A	Yes	No		
☐	☐	☐	1.	Cooler seals intact? (N/A if hand delivered) If no, document on CUR.
	☐	☐	2.	Coolers scanned for radiation. Is the reading ≤ to background levels? BKG CPM: ____ CPM Reading:_____
	☐	☐	3.	Chain of custody present? If no, document on CUR.
	☐	☐	4.	Bottles broken and/or are leaking? If yes, document on CUR.
	☐	☐	5.	Multiphasic samples obvious? If yes, document on CUR.
☐	☐	☐	6.	pH of all samples checked and meet requirements? If no, document on CUR.
	☐	☐	7.	Sufficient volume, proper container, and preservatives used for all analyses requested? (ref. Attachment 4 of SOP# DV-QA-0003).If no, document on CUR, and contact PM before proceeding.
	☐	☐	8.	Did chain of custody agree with label IDs and samples received? If no, document on CUR.
☐	☐	☐	9.	Were VOA samples without headspace? If no, document on CUR.
☐	☐	☐	10.	Were VOA vials preserved? Preservative ☐ HCl ☐ 4±2°C ☐ Sodium Thiosulfate ☐ Ascorbic Acid
☐	☐	☐	11.	Did the samples contain residual chlorine? If yes, document on CUR.
☐	☐	☐	12.	Sediment present in dissolved/filtered bottles? If yes, document on CUR.
☐	☐	☐	13.	Is sufficient volume provided for client requested MS, MSD or matrix duplicates? If no, document on CUR, and contact PM before proceeding.

Login Checks: ***Initials*** _____

	☐	☐	14.	Did the chain of custody include "received by" and "relinquished" by signatures, dates, and times?
☐	☐	☐	15.	Were special log in instructions read and followed?
☐	☐	☐	16.	Were AFCEE metals logged for refrigerated storage?
	☐	☐	17.	Were tests logged checked against the COC?
☐	☐	☐	18.	Was a Rush form completed for quick TAT?
☐	☐	☐	19.	Was a Short Hold form completed for any short holds?
	☐	☐	20.	Were special archiving instructions indicated in the General Comments? If so, what were they?

_____

Labeling and Storage Checks: ***Initials*** _____

☐	☐	☐	21.	Was the subcontract COC signed and sent with samples to bottle prep?
☐	☐	☐	22.	Did the sample ID, Date, and Time from label match the COC and what was logged?
☐	☐	☐	23.	Were stickers for special archiving instructions affixed to each box? See #21
☐	☐	☐	24.	Were AFCEE metals stored refrigerated?

Document any problems or discrepancies and the actions taken to resolve them on a Condition Upon Receipt Anomaly Report (CUR).

G:\QA\Edit\FORMS\Sample Receiving\Att 1_DV-QA-0003_Sample Receiving Checklist Rev 8.doc Revision 8.0 (1-26-11)

SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g., Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, evaporation, and drying. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	Used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above ½ the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank [1]	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	Also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (**TNI**)
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.3.1 **Negative Controls for Microbiological Methods** – Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are*:*

Table 24-2. Negative Controls for Microbiology

Control Type	Details
Sterility Checks (Media)	Analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
Filtration Blanks	Blanks that are run at the beginning and end for each sterilized filtration unit used in a filtration series. For pre-sterilized single use funnels a sterility check is performed on at least one funnel per lot.
Sterility checks (Sample Containers)	Performed on at least one container per lot of purchased, pre-sterilized containers. If containers are prepared and sterilized by the laboratory, one container per sterilization batch is checked. Container sterility checks are performed using non-selective growth media.
Sterility Checks (Dilution Water)	Performed on each batch of dilution water prepared by the laboratory and on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.
Sterility Checks (Filters)	Also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms.

Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However,

in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.4.2 Positive Controls for Microbiological Methods

- Each lot of pre-prepared media (including chromofluorogenic reagent) and each batch of laboratory prepared media is tested with a pure culture of known positive reaction.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects.If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.

Table 24-3. Sample Matrix Control

Control Type	Details	
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on a semi-annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically

derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.

- If either the high or low end of the control limit changes by $\leq$ 5% from previous, the control chart is visually inspected and, using professional judgment, the limit(s) may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to Denver SOP DV-QA-003P, *Quality Assurance Program* for a detailed description of the control charting procedure.

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with a LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.

- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI and Department of Defense (DoD) work, there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

- Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be

located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.

Though marginal exceedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.3 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.4 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additonal Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.
-

SECTION 25. REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation

requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

25.2.2 Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Reporting Limits

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 Non-accredited tests shall be clearly identified in the case narrative when claims of accreditation to the TNI standard are made.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above. TestAmerica Denver rarely utilizes this report.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. The Denver laboratory offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, SEDD 2A, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific

electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 Client Confidentiality

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-800-765-0980 (or for e-mails: please notify us immediately by e-mail or by phone (1-800-765-0980) and delete this material from any computer).

25.7 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the job number followed by "Rev#".

When the report is re-issued, a notation of "revised report "is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 11/3/08 to include toluene in sample NQA1504 per client's request.*

25.9 Policies on Client Requests for Amendments

25.9.1 Policy on Data Omissions or Reporting Limit Increases

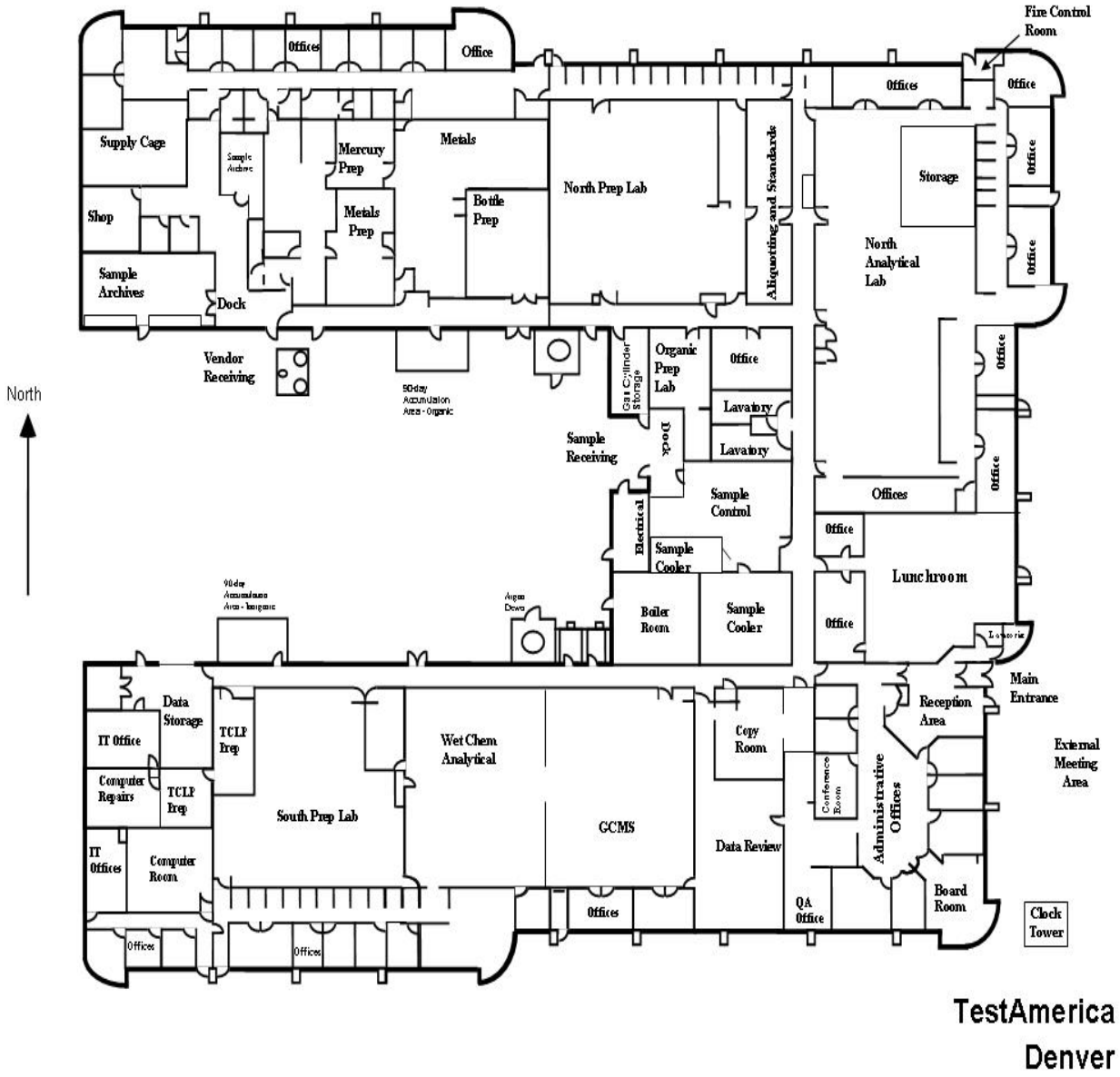
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. Laboratory Floor Plan



Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)

Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Aliquot, aliquant: A measured portion of a sample taken for analysis.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

LOD Verification [a.k.a., MDL Verification]: A processed QC sample in the matrix of interest, spiked with the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests and processed through the entire analytical procedure.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. (TNI)

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICP/MS – ICP/Mass Spectrometry
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification check standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control

QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3. Laboratory Certifications, Accreditations, Validations

TestAmerica Denver maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Lab ID	Program	Authority
2907.01	DoD ELAP	A2LA
	ISO/IEC 17025	A2LA
E87667	NELAC Primary AB	Florida
200017	NELAC Secondary AB	Illinois
E-10166	NELAC Secondary AB	Kansas
30785	NELAC Secondary AB	Louisiana
	NELAC Secondary AB	New Hampshire
	NELAC Secondary AB	New Jersey
11964	NELAC Secondary AB	New York
CO200001	NELAC Secondary AB	Oregon
68-00664	NELAC Secondary AB	Pennsylvania
T104704183-08-TX	NELAC Secondary AB	Texas
QUAN5	NELAC Secondary AB	Utah
	State Program	Alabama
UST-30	State Program	Alaska
AZ0713	State Program	Arizona
88-0687	State Program	Arkansas
2513	State Program	California
N/A	State Program	Colorado
486-03	State Program-RAM license	Colorado
PH-0686	State Program	Connecticut
N/A	State Program	Georgia
CO00026	State Program	Idaho
370	State Program	Iowa
CO0002	State Program	Maine
268	State Program	Maryland
8-999-405	State Program	Minnesota
CO0026	State Program	Nevada
N/A	State Program	New Mexico
358	State Program	North Carolina
R-034	State Program	North Dakota
8614	State Program	Oklahoma
72002	State Program	South Carolina
TN02944	State Program	Tennessee
C1284	State Program	Washington
354	State Program	West Virginia
999615430	State Program	Wisconsin
P330-08-00036	USDA	

The certificates and parameter lists (which may differ) are available, upon request, from a laboratory representative. for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

Appendix F

Laboratory Qualifications

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Karen Kuoppala Phone: 303-736-1203
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2013 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the current DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Metals			
Aluminum	----------	EPA 6010B/6010C	EPA 6010B/6010C
Antimony	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Arsenic	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Barium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Beryllium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Boron	----------	EPA 6010B/6010C	EPA 6010B/6010C
Cadmium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Calcium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Chromium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Cobalt	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Copper	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A

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Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Iron	----------	EPA 6010B/6010C	EPA 6010B/6010C
Lead	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Lithium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Magnesium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Manganese	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Mercury	----------	EPA 7470A	EPA /7471A/7471B
Molybdenum	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Nickel	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Potassium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Selenium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Silica	----------	EPA 6010B/6010C	EPA 6010B/6010C
Silicon	----------	EPA 6010B/6010C	EPA 6010B/6010C
Silver	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Sodium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Strontium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Thallium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Tin	----------	EPA 6010B/6010C	EPA 6010B/6010C
Titanium	----------	EPA 6010B/6010C	EPA 6010B/6010C
Vanadium	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Zinc	----------	EPA 6010B/6010C/6020/6020A	EPA 6010B/6010C/6020/6020A
Nutrients			
Nitrate (as N)	By calculation	By calculation/ EPA 9056/9056A	By calculation/ EPA 9056/9056A
Nitrate-nitrite (as N)	EPA 353.2	EPA 353.2/ EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	SM 4500-NO2 B	SM 4500-NO2 B/ EPA 9056/9056A	EPA 9056/9056A
Orthophosphate (as P)	----------	EPA 9056/9056A	EPA 9056/9056A
Total phosphorus	----------	EPA 6010B/6010C	EPA 6010B/6010C
Demands			
Total organic carbon	----------	EPA 9060 /9060A	EPA 9060 /9060A
Total organic halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity	SM 2320 B	SM 2320 B	SM 2320 B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	----------	EPA 9056/9056A	EPA 9056/9056A
Total organic carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Chloride	----------	EPA 9056/9056A	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Conductivity	----------	EPA 9050/EPA 9050A	EPA 9050/EPA 9050A
Cyanide	----------	EPA 9010B/9012A/9012B	EPA 9010B/9012A/9012B
Ferrous Iron	SM 3500 Fe B, D	SM 3500 Fe B, D	
Fluoride	----------	EPA 9056/9056A	EPA 9056/9056A
Hexavalent Chromium	EPA 7196A	EPA 7196A	EPA 7196A/3060A
pH	----------	EPA 9040B/9045C	EPA 9040B/9045C
Oil and Grease (HEM and SGT-HEM)	EPA 1664A	EPA 1664A	9071B
Percent moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	EPA 9066
Solids, Total	SM 2540 B	SM 2540 B	SM 2540 B
Solids, Total Suspended	SM 2540 D	SM 2540 D	SM 2540 D
Solids, Total Dissolved	SM 2540 C	SM 2540 C	SM 2540 C
Sulfate	----------	EPA 9038/9056/9056A	EPA 9038/9056/9056A
Sulfide, Total	----------	EPA 9034	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	----------
Purgeable Organics (volatiles)			
Acetone	----------	EPA 8260B	EPA 8260B
Acetonitrile	----------	EPA 8260B	EPA 8260B
Acrolein	----------	EPA 8260B	EPA 8260B
Acrylonitrile	----------	EPA 8260B	EPA 8260B
Allyl Chloride	----------	EPA 8260B	EPA 8260B
Benzene	----------	EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
Bromobenzene	----------	EPA 8260B	EPA 8260B
Bromochloromethane	----------	EPA 8260B	EPA 8260B
Bromodichloromethane	----------	EPA 8260B	EPA 8260B
Bromoform	----------	EPA 8260B	EPA 8260B
Bromomethane	----------	EPA 8260B	EPA 8260B
2-Butanone	----------	EPA 8260B	EPA 8260B
n-Butyl alcohol	----------	EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
n-Butylbenzene	----------	EPA 8260B	EPA 8260B
Sec-Butylbenzene	----------	EPA 8260B	EPA 8260B
Tert-Butylbenzene	----------	EPA 8260B	EPA 8260B
Carbon disulfide	----------	EPA 8260B	EPA 8260B
Carbon tetrachloride	----------	EPA 8260B	EPA 8260B
Chlorobenzene	----------	EPA 8260B / 8021B	EPA 8260B / 8021B
2-Chloro-1,3-butadiene	----------	EPA 8260B	EPA 8260B
Chloroethane	----------	EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	----------	EPA 8260B	EPA 8260B
Chloroform	----------	EPA 8260B	EPA 8260B
1-Chlorohexane	----------	EPA 8260B	EPA 8260B
Chloromethane	----------	EPA 8260B	EPA 8260B
Chloroprene	----------	EPA 8260B	EPA 8260B
3-Chloroprene	----------	EPA 8260B	EPA 8260B
4-Chlorotoluene	----------	EPA 8260B	EPA 8260B
2-Chlorotoluene	----------	EPA 8260B	EPA 8260B
Cyclohexane	----------	EPA 8260B	EPA 8260B
Cyclohexanone	----------	EPA 8260B	EPA 8260B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dibromochloromethane	----------	EPA 8260B	EPA 8260B
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504	EPA 504/ EPA 8260B/8011	EPA 8260B/8011
Dibromochloromethane	----------	EPA 8260B	EPA 8260B
Dichlorodifluoromethane	----------	EPA 8260B	EPA 8260B
Dibromomethane	----------	EPA 8260B	EPA 8260B
1,2 Dibromoethane (EDB)	EPA 504	EPA 504/ EPA 8260B/8011	EPA 8260B/8011
1,2-Dichlorobenzene	----------	EPA 8260B/8021B	EPA 8260B/8021B
1,3-Dichlorobenzene	----------	EPA 8260B/8021B	EPA 8260B/8021B
1,4-Dichlorobenzene	----------	EPA 8260B/8021B	EPA 8260B/8021B
cis-1,4-Dichloro-2-butene	----------	EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-butene	----------	EPA 8260B	EPA 8260B
1,1-Dichloroethane	----------	EPA 8260B	EPA 8260B
1,2-Dichloroethane	----------	EPA 8260B	EPA 8260B
1,1-Dichloroethene	----------	EPA 8260B	EPA 8260B
1,2-Dichloroethene	----------	EPA 8260B	EPA 8260B
cis-1,2-Dichloroethene	----------	EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene	----------	EPA 8260B	EPA 8260B
Dichlorofluoromethane	----------	EPA 8260B	EPA 8260B
1,2-Dichloropropane	----------	EPA 8260B	EPA 8260B
1,3-Dichloropropane	----------	EPA 8260B	EPA 8260B
2,2-Dichloropropane	----------	EPA 8260B	EPA 8260B
1,1-Dichloropropene	----------	EPA 8260B	EPA 8260B
1,3-Dichloropropene	----------	EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene	----------	EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene	----------	EPA 8260B	EPA 8260B
Diethyl ether	----------	EPA 8260B	EPA 8260B
Di-isopropylether	----------	EPA 8260B	EPA 8260B
1,4-Dioxane	----------	EPA 8260B/8260B SIM	EPA 8260B/8260B SIM
Ethanol	----------	EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
Ethyl acetate	----------	EPA 8260B	EPA 8260B
Ethyl benzene	----------	EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
Ethyl methacrylate	----------	EPA 8260B	EPA 8260B
Ethylene Glycol	----------	EPA 8015C	EPA 8015C
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/AK101/8015D	EPA 8015B/8015C/AK101/8015D
Hexane	----------	EPA 8260B	EPA 8260B
2-Hexanone	----------	EPA 8260B	EPA 8260B
Hexachlorobutadiene	----------	EPA 8260B	EPA 8260B
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8015B/8015C	EPA 8260B/8015B/8015C
Isopropyl alcohol	----------	EPA 8260B	EPA 8260B
Isopropylbenzene	----------	EPA 8260B	EPA 8260B
1,4-Isopropyltoluene	----------	EPA 8260B	EPA 8260B
Iodomethane	----------	EPA 8260B	EPA 8260B
Methacrylonitrile	----------	EPA 8260B	EPA 8260B
Methanol	----------	EPA 8015B/8015C	EPA 8015B/8015C
Methyl acetate	----------	EPA 8260B	EPA 8260B
Methyl cyclohexane	----------	EPA 8260B	EPA 8260B
Methylene chloride	----------	EPA 8260B	EPA 8260B
Methyl ethyle ketone (MEK)	----------	EPA 8260B	EPA 8260B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Methyl isobutyl ketone	----------	EPA 8260B	EPA 8260B
Methyl methacrylate	----------	EPA 8260B	EPA 8260B
Methyl tert-butyl ether (MtBE)	----------	EPA 8260B/8021B	EPA 8260B/8021B
4-Methyl-2-pentanone	----------	EPA 8260B	EPA 8260B
Naphthalene	----------	EPA 8260B	EPA 8260B
2-Nitropropane	----------	EPA 8260B	EPA 8260B
2,2' Oxybisethanol	----------	EPA 8015C	EPA 8015C
2-Pentanone	----------	EPA 8260B	EPA 8260B
2-Propanol	----------	EPA 8260B	EPA 8260B
Propionitrile	----------	EPA 8260B	EPA 8260B
n-Propylbenzene	----------	EPA 8260B	EPA 8260B
Propylene Glycol	----------	EPA 8015C	EPA 8015C
Styrene	----------	EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	----------	EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	----------	EPA 8260B	EPA 8260B
Tetrachloroethene	----------	EPA 8260B	EPA 8260B
Tetrahydrofuran	----------	EPA 8260B	EPA 8260B
Toluene	----------	EPA 8260B / 8021B/AK101	EPA 8260B / 8021B/AK101
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	EPA 1664A	
1,2,3-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	----------	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	----------	EPA 8260B	EPA 8260B
Trichloroethene	----------	EPA 8260B	EPA 8260B
Trichlorofluoromethane	----------	EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	----------	EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	EPA 504.1	EPA 504.1/ EPA 8260B/8011	EPA 8260B/8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B	EPA 8260B
Triethylene Glycol	----------	EPA 8015C	EPA 8015C
1,2,3-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
1,2,4-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	----------	EPA 8260B	EPA 8260B
Vinyl acetate	----------	EPA 8260B	EPA 8260B
Vinyl chloride	----------	EPA 8260B	EPA 8260B
Xylenes, total	----------	EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
1,2-Xylene	----------	EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
M+P-Xylene	----------	EPA 8260B/8021B/AK101	EPA 8260B/8021B/AK101
Methane	----------	RSK-175	----------
Ethane	----------	RSK-175	----------
Ethylene (Ethene)	----------	RSK-175	----------
Acetylene	----------	RSK-175	----------
Acetylene Ethane	----------	RSK-175	----------
Extractable Organics (semivolatiles)			
Acenaphthene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Acenaphthylene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Acetophenone	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alachlor	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Anthracene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Aramite	----------	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde	----------	EPA 8270C/8270D	EPA 8270C/8270D
Benzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	----------	EPA 8270C/8270D	EPA 8270C/8270D
Benzo (a) anthracene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Benzo (b) fluoranthene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Benzo (k) fluoranthene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Benzo (ghi) perylene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Benzo (a) pyrene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Benzyl alcohol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroethoxy) methane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroethyl) ether	----------	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-chloroisopropyl) ether (2,2'Oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
Bis (2-ethylhexyl) phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	----------	EPA 8270C/8270D	EPA 8270C/8270D
Butyl benzyl phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-sec-Butyl-4,6-dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloro-3-methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	----------	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Cresols	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diallate	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dibenzo (a,h) anthracene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Dibenzofuran	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dichlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alpha-,alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Di-n-butyl phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Di-n-octyl phthalate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dinitrotoluene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dioxane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C, AK102, TX 1005/8015D	EPA 8015B/8015C, AK102, TX 1005/8015D
Ethyl methanesulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	----------	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Fluorene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Gasoline Range Organics	----------	TX 1005	TX 1005
Hexachlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Isodrin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	----------	EPA 8270C/8270D	EPA 8270C/8270D
Methapyrilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
2-Methyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Methyl methane sulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Methylnaphthalene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
2-Methylnaphthalene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
2-Methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
1,4-Naphthoquinone	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
5-Nitro-o-toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, methyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	----------	EPA 8270C/8270D/8321A/8321B	EPA 8270C/8270D/8321A/8321B
Phenacetin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Phenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Phenylenediamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phorate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	----------	EPA 8270C/8270D/8270SIM	EPA 8270C/8270D/8270SIM
Pyridine	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Safrole	----------	EPA 8270C/8270D	EPA 8270C/8270D
Sulfotepp	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin	----------	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-Triethyl phosphorothioate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Tris(2,3-Dibromopropyl) phosphate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C, AK103	EPA 8015B/8015C, AK103
Pesticides/Herbicides/PCBs			
Aldrin	----------	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC	----------	EPA 8081A/8081B	EPA 8081A/8081B
Beta-BHC	----------	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	----------	EPA 8081A/8081B	EPA 8081A/8081B
Gamma-BHC	----------	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
Alpha-Chlordane	----------	EPA 8081A/8081B	EPA 8081A/8081B
Gamma-Chlordane	----------	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	----------	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8081A/8081B/8141A/8141B	EPA 8081A/8081B/8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4-D	----------	EPA 8151A/8321A	EPA 8151A/8321A
Dalapon	----------	EPA 8151A/8321A	EPA 8151A/8321A
2,4-DB	----------	EPA 8151A/8321A	EPA 8151A/8321A
4,4'-DDD	----------	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	----------	EPA 8081A/8081B	EPA 8081A/8081B
4,4',-DDT	----------	EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A/8321A	EPA 8151A/8321A
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop	----------	EPA 8151A/8321A	EPA 8151A/8321A
Dicofol	----------	EPA 8081A/8081B	EPA 8081A/8081B
Dieldrin	----------	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb	----------	EPA 8151A/8321A	EPA 8151A/8321A
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I	----------	EPA 8081A/8081B	EPA 8081A/8081B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Endosulfan II	----------	EPA 8081A/8081B	EPA 8081A/8081B
Endonsulfan sulfate	----------	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	----------	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	----------	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	----------	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor	----------	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	----------	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A/8321A	EPA 8151A/8321A
MCPP	----------	EPA 8151A/8321A	EPA 8151A/8321A
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	----------	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	----------	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	----------	EPA 8082/8082A	EPA 8082/8082A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8081A/8081B/8141A/8141B	EPA 8081A/8081B/8141A/8141B
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T	----------	EPA 8151A/8321A	EPA 8151A/8321A
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP	----------	EPA 8151A/8321A	EPA 8151A/8321A
Toxaphene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-triethylphos phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1,3-Dinitrobenzene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
3,5-Dinitroaniline	----------	8330B	8330B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
2-Amino-4,6-dinitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
4-Amino-2,6-dinitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Nitrobenzene	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Octahydro-1,3,5,7-tetrabitro-1,3,5,7-tetrazocine (HMX)	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Tetryl (methyl2,4,6-trinitrophenylnitramine	----------	EPA 8330A/8330B/8321A/8321B	EPA 8330A/8330B/8321A/8321B
Hydrazines			
Hydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
Monomethyl hydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
1,1-Dimethylhydrazine	SOP DV WC-0077	SOP DV WC-0077	SOP DV WC-0077
Perfluorinated Hydrocarbons (PFCs) and Perfluorinated Sulfonates (PFSs)			
Perfluorobutanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoropentanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroheptanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorononanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluoroundecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorododecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Perfluorotridecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorotetradecanoic acid	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorobutane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorohexane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorodecane Sulfonate	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
Perfluorooctane Sulfonamide	SOP DV-LC-0012	SOP DV-LC-0012	SOP DV-LC-0012
N-Nitrosodimethylamine (NDMA)	SOP DV-LC-0019	SOP DV-LC-0019	SOP DV-LC-0019
Hazardous Waste Characteristics			
Conductivity	----------	EPA 9050A	EPA 9050A
Corrosivity	----------	EPA 9040B	9045C
Ignitibility	EPA 1010/EPA 1010A	EPA 1010/EPA 1010A	EPA 1010/EPA 1010A
Paint Filter Liquids Test		EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
ToxicityCharacteristic Leaching Procedure	----------	EPA 1311	EPA 1311
Organic Prep Methods			
Separatory Funnel Liquid-Liquid Extraction	----------	EPA 3510C	----------
Continuous Liquid-Liquid Extraction	----------	EPA 3520C	----------
Soxhlet Extraction	----------	----------	EPA 3540C
Microwave Extraction	----------	----------	EPA 3546
Ultrasonic Extraction	----------	----------	EPA 3550B
Ultrasonic Extraction	----------	----------	EPA 3550C
Waste Dilution	----------	EPA 3580A	EPA 3580A
Solid Phase Extraction Volatiles Purge and trap Volatiles purge and trap for soils	----------	EPA 3535A EPA 5030B	EPA 5030B EPA 5035
Organic Cleanup Procedures			
Florisil Cleanup	----------	EPA 3620B	EPA 3620B
Florisil Cleanup	----------	EPA 3620C	EPA 3620C
Sulfur Cleanup	----------	EPA 3660B	EPA 3660B
Sulfuric Acid/Permanganate Cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid Digestion Total Recoverable or Dissolved Metals	----------	EPA 3005A	----------
Acid Digestion for Total Metals	----------	EPA 3010A	----------

Parameter/Analyte	Non-Potable Water	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Acid Digestion for Total Metals	----------	EPA 3020A	----------
Acid Digestion of Sediments, Sludges and Soils	----------	----------	EPA 3050B

The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the current DoD Quality Systems Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

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Presented this 5th day of October 2011.

President & CEO
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2013

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

DEPARTMENT OF THE ENVIRONMENT
WATER SUPPLY PROGRAM

Certifies That

TestAmerica DENVER
4955 Yarrow Street, Arvada, CO 80002

Having duly met the requirements of the
Regulations Governing Laboratory Certification
And Standards of Performance In Accordance With
The Annotated Code of Maryland,
is hereby approved as a

State Certified Water Quality Laboratory

To perform the analyses indicated on the Annual Certified Parameter List,
which must accompany this certificate.

Certification # 268

Date Issued February 2, 2012

Expiration Date March 31, 2013
(Not Transferable)

Administrator, Water Supply Program

This certification is subject to unannounced laboratory inspections

CONSPICUOUSLY DISPLAY IN THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST. MDE00768

Issued Date: February 3, 2012
Expiration Date: March 31, 2013

MDE
MARYLAND DEPARTMENT OF THE ENVIRONMENT
1800 Washington Boulevard • Baltimore MD 21230
410-537-3000 • 1-800-633-6101

Martin O'Malley
Governor

Anthony G. Brown
Lieutenant Governor

Robert M. Summers, Ph.D.
Secretary

SDWA ANNUAL CERTIFIED PARAMETER LIST

TestAmerica - Denver
4955 Yarrow Street
Arvada, CO 80002

Robert C. Hanisch
Certificate # 268
EPA ID # CO00026

ANALYTE	METHOD	STATUS
Antimony	EPA 200.8, Rev. 5.4	Certified
Arsenic	EPA 200.8, Rev. 5.4	Certified
Barium	EPA 200.7, Rev. 4.4	Certified
Barium	EPA 200.8, Rev. 5.4	Certified
Beryllium	EPA 200.7, Rev. 4.4	Certified
Beryllium	EPA 200.8, Rev. 5.4	Certified
Cadmium	EPA 200.7, Rev. 4.4	Certified
Cadmium	EPA 200.8, Rev. 5.4	Certified
Chromium	EPA 200.7, Rev. 4.4	Certified
Chromium	EPA 200.8, Rev. 5.4	Certified
Copper	EPA 200.7, Rev. 4.4	Certified
Copper	EPA 200.8, Rev. 5.4	Certified
Dibromochloropropane (DBCP)	EPA 504.1, Rev. 1.1	Certified
Ethylene Dibromide (EDB)	EPA 504.1, Rev. 1.1	Certified
Fluoride	EPA 300.0, Rev. 2.1	Certified
Lead	EPA 200.8, Rev. 5.4	Certified
Mercury	EPA 245.1, Rev. 3.0	Certified
Nitrate	EPA 300.0, Rev. 2.1	Certified
Nitrate	EPA 353.2, Rev. 2.0	Certified
Nitrite	EPA 300.0, Rev. 2.1	Certified
Nitrite	EPA 353.2, Rev. 2.0	Certified
Selenium	EPA 200.8, Rev. 5.4	Certified
Thallium	EPA 200.8, Rev. 5.4	Certified
Uranium	EPA 200.8, Rev. 5.4	Certified

Appendix G

Regulatory Comments and Responses

Response to Comments for the
Draft UFP-QAPP at CS-C503
Performance-Based Restoration
Joint Base Andrews Naval Air Facility Washington, Camp Springs, Maryland
July 2012

General Clarification

To clarify the scope of the sampling and analysis proposed in the UFP-QAPP for the Phase I RI at CS-C503, a summary of the site history; surface water and stormwater drainage area; contaminants of potential concern (COPCs); and proposed sampling approach has been added as a preface to these Response to Comments (RTCs). This summary provides additional clarification and justification for the scope of the project and supports the RTCs provided.

Site History

A 50-foot by 500-foot stormwater retention pond and associated storm sewer was constructed near the intersection of Arnold Avenue and North Perimeter Road and northeast of Building 1889 on Joint Base Andrews Naval Air Facility Washington (JBA). The retention pond was constructed between the years 1990 and 1995 based on an aerial photograph review to serve the Base Exchange (BX, Building 1811) site. In 2007, maintenance activities at the retention pond were completed, including the excavation of 870 tons of sediment over a 220-foot by 35-foot area. Two waste characterization samples were collected from sediment stockpiles and analyzed in accordance with the landfill disposal permit requirements. The sediment samples were analyzed for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, herbicides, metals, mercury, total petroleum hydrocarbons (TPH), cyanide, sulfides, pH, and ignitability. Results of the analysis indicated that PCBs were present at concentrations of 190 parts per billion (ppb) and 300 ppb. In addition, TPH (as Diesel Range Organics) was detected at concentrations of 120 parts per million (ppm) and 640 ppm. The waste profile was approved by the Maryland Department of the Environment (MDE) as non-hazardous.

While the PCB-contaminated sediment within the pond excavation limits was removed in 2007, CS-C503 was designated in the Environmental Restoration Program (ERP) at JBA as a result of the 2007 waste characterization PCB-detection. In the 2011 Federal Facility Agreement (FFA) between the United States Air Force (USAF) and the United States Environmental Protection Agency (USEPA), CS-C503 was identified as a removal action site under Section 6.7.4 and under 6.7.4.2 it stated that the source and extent of PCB contamination is unknown. Therefore, the scope of the Phase I Remedial Investigation (RI) is to investigate the source and extent of PCB-contamination prior to initiating a removal action if residual contamination is identified.

Surface Water and Stormwater Drainage Areas

Prior to submitting the Draft UFP-QAPP, the drainage area to the CS-C503 Retention Pond was mapped by a utility locating subcontractor on 6 June 2012 to define the investigation extents. The Retention Pond drainage area originates from the BX parking lot and surrounding grass areas. Runoff from the building roof drains, asphalt loading area, adjacent asphalt parking lot, and grass areas surrounding the BX is conveyed east and north via the storm sewer within the west boulevard of Arnold Avenue. The storm sewer eventually outlets at the south end of the CS-C503 Retention Pond (as designated on **Figure 4** in blue). The 6 June 2012 survey confirmed that no other areas drain into the CS-C503 Retention Pond other than overland drainage in the near vicinity of the pond (extent of surface drainage area is shaded in **Figure 5**).

The following areas do not contribute stormwater to the CS-C503 Retention Pond:

- The Club at Andrews (Building 1889);
- The Club at Andrews Parking Lot;
- The electrical substation (Building 1870);
- The detention pond south of The Club at Andrews;
- The Arnold Avenue roadway and boulevard drainage; and
- The North Perimeter Road roadway and ditches.

With the exception of North Perimeter Road, these areas are captured and conveyed to the storm sewer within the Arnold Avenue west curb line (as designated on **Figure 4** in green). The Arnold Avenue storm sewer is not connected to the CS-C503 Retention Pond or the storm sewer from the BX to the CS-C503 Retention Pond. Runoff from North Perimeter Road flows via an open channel ditch and crosses North Perimeter Road to the northwest via a culvert.

The construction of the electrical substation, Building 1870, built between the years 1964 and 1974 was originally noted as a potential contaminant source because transformers have historically contained PCBs. However, The Club at Andrews, Building 1889, is situated between the electrical substation and the PCB-contaminated retention pond. The Club at Andrews' storm sewer would intercept potentially contaminated sediment/stormwater from the transformer site and direct it to the Arnold Avenue storm sewer that is not connected to the CS-C503 Retention Pond. In addition, the

aerial imagery review confirmed that The Club at Andrews pre-dates the electrical transformer; therefore, at no point was there a direct transport pathway between the electrical transformer and the CS-C503 Retention Pond.

Contaminants of Potential Concern

The FFA states that *"PCBs were discovered in the excavated sediment, which were removed and disposed off-site. The source of the PCBs and the extent of PCB contamination remaining, if any, are unknown"*. The COPCs identified in the UFP-QAPP for the Phase I RI at CS-C503 were based on the FFA, Tier I Partnering Meeting discussions, Tier I Meeting minutes and previous sampling results during the 2007 Retention Pond Maintenance project. Based on this information PCBs were determined to be the primary COPC. DRO and GRO were also included as PCBs and are commonly present in petroleum-based oils. In addition, if PCBs are detected above the screening criteria in the soil/sediment samples collected, one sample with the highest concentration of PCBs will be analyzed for dioxins/furans. These chemicals can be associated with PCB manufacturing (as a chemical byproduct). In addition to the use of PCBs within transformers, PCBs are sometimes oxidized to create dioxins and furans.

Proposed Sampling Approach

The Phase I RI scope consists of an evaluation of whether the COPCs are present or absent within:

- The CS-C503 Retention Pond; and
- The associated stormwater drainage area (defined by the 6 June 2012 utility survey).

The proposed sample locations and matrices have been expanded based on the USEPA Comments to the Draft UFP-QAPP and are provided on the revised **Figure 4**. The following table summarizes the matrix by location (sample locations are identified on **Figure 4**):

Sample Location	Sample ID per Matrix			
	Sediment	Soil	Stormwater	Surface Water
01		CSC503-01-SB-001		
02		CSC503-02-SB-002		
03		CSC503-03-SB-003		
04		CSC503-04-SB-004		
05		CSC503-05-SB-005		
06				CSC503-06-SW-006
07	CSC503-07-SD-007			
08	CSC503-08-SD-008			
09		CSC503-09-SB-009		
10	CSC503-10-SD-010			
11	CSC503-11-SD-011			
12				CSC503-12-SW-012
13		CSC503-13-SB-013		
14	CSC503-14-SD-014			
15				CSC503-15-SW-015
16		CSC503-16-SB-016		
17	CSC503-17-SD-017[a]		CSC503-17-ST-018[b]	
18	CSC503-18-SD-019[c]	CSC503-18-SB-020[c]	CSC503-18-ST-021[b]	
19	CSC503-19-SD-022[c]	CSC503-19-SB-023[c]	CSC503-19-ST-024[b]	
20	CSC503-20-SD-025[c]	CSC503-20-SB-026[c]	CSC503-20-ST-027[b]	
21	CSC503-21-SD-028[c]	CSC503-21-SB-029[c]	CSC503-21-ST-030[b]	
22	CSC503-22-SD-031[a]		CSC503-22-ST-032[b]	
23	CSC503-23-SD-033[a]		CSC503-23-ST-034[b]	

[a] A sediment sample will be collected if enough sediment is present within the designated catch basin (CB) at the time of sampling.
[b] A stormwater sample will be collected if enough stormwater is present at the time of sampling.
[c] A sediment sample will be collected if enough sediment is present within the designated CB at the time of sampling. If sediment is not present in the CB, a soil sample will be collected from the ground surface adjacent to the CB.

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Comment #	Page	Section/ Paragraph/ Line No.	Comment	A, D, E, FD or X[1]	Response	A or D[2]
MDE (Rick Grills) – Comments Received: 11 July 2012						
1	3-1	Wksht #3	In the column titled "E-Mail and Mailing Address", please make the following change in the mailing address for Rick Grills. Change "Hazardous Waste Program" to "Land Restoration Program."	A	Page 3-1, Distribution List, the mailing address for Rick Grills has been changed.	
2	9-1	Wksht #9	In the column titled "Phone #", please change the phone number for Rick Grills from "(410) 631-3398" to "(410) 537-3398."	A	Page 9-1, Project Scoping Session Participants Sheet, the phone number for Rick Grills has been changed.	
3	11-3	Wksht #11	This document states that "MDE Interim Final Cleanup Standards for GRO/DRO (MDE 2008) will be used as human health screening criteria." These Maryland Department of Environment (MDE) cleanup standards were specifically developed to support the MDE's Voluntary Cleanup Program and were intended to be used in-lieu of a formal risk assessment. These MDE standards are based on a cancer risk of 10E-5, and are less stringent than the risk-based standards used by EPA Region III; which are based on a more conservative theoretical cancer risk of 10E-6. MDE considers these cleanup standards as "To Be Considered (TBC)" for Comprehensive Environmental Response, Compensation and Liability (CERCLA) activities at Joint Base Andrews.	A	Page ES-2, Executive Summary, the following sentence was revised: GRO/DRO analytical results will be compared to the June 2008 Maryland Department of the Environment (MDE) Interim Final Cleanup Standards as no USEPA Regional Screening Levels (RSLs) are listed for comparison. Page 11-3, Worksheet #11, Project Quality Objectives/Systematic Planning Process Statements, Step 5, Analytical Approach, the following sentence was added after the first paragraph following the bullets: MDE cleanup standards for GRO/DRO are intended to be used in conjunction with a screening level risk assessment even though MDE considers these cleanup standards as "To Be Considered" (TBC) as no USEPA screening criteria exist.	

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4	15-1	Wksht #15	Please reference and footnote appropriately all text and tables in this report relating to the use of "MDE Interim Final Cleanup Standards (MDE 2008)" as risk-based cleanup criteria. MDE wishes the use of these cleanup standards to be on a "TBC" basis, and not as the primary risk-based cleanup criteria at CERCLA sites.	A	Page 15-2, Worksheet #15, Reference Limits and Evaluation Table, the footnotes referring to MDE cleanup standards were revised as follows: The Screening Criteria Reference is taken from the MDE Interim Final Cleanup Standards (MDE 2008). MDE considers the use of these cleanup standards to be on a TBC basis, and not the primary-based cleanup criteria at CERCLA sites.	
USEPA-CRL (Jay Burman) – Comments Received: 1 October 2012 (Comments compiled by Andrew Sochanski, USEPA RPM)						
1		General	a) Water often acts as a transport mechanism for contamination. As such, the reviewer strongly recommends sampling and analyzing sediment and water samples from CS-C503 Retention Pond Storm Water System. Storm water system sample analysis (both water and sediment) could prove to be a very useful tool in determining the migration pathway of the contamination.	a) A b) A c) A d) E	a) Surface water samples will be collected from Sample Locations 6, 12, and 15 located at the inlet, midpoint, and outlet of the pond, respectively. In addition, if stormwater is available at the time of sampling, stormwater samples will be collected from the catch basins located at Sample Locations 17 through 23. See the sample summary table located on Page 2 of 12 of these RTCs for a detailed list of the proposed Sample ID as well as the matrices proposed for analysis at each location. With the addition of the proposed surface water and stormwater analyses, references to each analysis have been added to the following worksheets: • Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements; • Worksheet #12 – Measurement Performance Criteria Tables;	

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Comment #	Page	Section/ Paragraph/ Line No.	Comment	A, D, E, FD or X[1]	Response	A or D[2]
					• Worksheet #14 – Summary of Project Tasks; • Worksheet #15 – Reference Limits and Evaluation; • Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table; • Worksheet #19 – Analytical SOP Requirements Table; • Worksheet #20 – Field Quality Control Sample Summary Table; • Worksheet #21 – Project Sampling SOP References Table; • Worksheet #23 – Analytical Laboratory SOP References Table • Worksheet #27 – Sample Custody Requirements Table; • Worksheet #28 – Analytical Laboratory QC Samples; • Worksheet #30 – Analytical Services Table; and • Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table.	
			b) Varying concentrations of contamination, if present, throughout the storm water system would aid in pin pointing the location of the source of contamination. Sampling should begin at the first storm water inlet southwest of the Burger King parking lot; the two inlets north of G Street and the inlet at the northeast corner of Base Exchange Building 1811.		b) Sediment samples from storm sewer catch basins located within grass areas designated as Sample Locations 21, 22, and 23 were added as requested. If sediment volume is insufficient at the time of sampling, then soil samples adjacent to the catch basins will be collected as an alternative.	
			c) The influent into CS-C503 as well as the effluent should be sampled and analyzed as well. Water and sediment from the overflow structure should be sampled and analyzed.		c) See response to Comment #1a for the addition of surface water samples. A sediment sample at the effluent structure was previously proposed and therefore, no	

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					sediment sampling change is included at this time. d) As detailed in the Surface Water and Stormwater Drainage Areas section of Page 1 of 12 of these RTCs, the Arnold Avenue Stormwater System (as designated on **Figure 4** in green) is not connected to the CS-C503 Retention Pond Stormwater System (as designated on **Figure 4** in blue). The two stormwater systems are independent of each other as confirmed on 6 June 2012 by the subsurface utility subcontractor. The additional sample areas east of Westover Drive and again southeast of The Club at Andrews Building 1889 have not been included in this investigation. Those areas are captured by catch basins and routed to the Arnold Avenue Stormwater System as noted on **Figure 4**.	
			d) Sampling the storm water vault (sediment sample SD04) is an excellent idea. However, sampling water and sediment from the Arnold Avenue Storm water System at the points before it perpendicularly crosses the CS-C503 Retention Pond Storm Water System east of Westover Drive and again southeast of The Club at Andrews Building 1889 would aid in identifying any contamination that may be coming from east of Arnold Avenue.			
2		General	A water contaminant level baseline needs to be established on the water in CS-C503 Retention Pond. It is recommended that a minimum of three water samples be collected and analyzed for the full suite of analytes from the pond; one near the influent, one near the effluent, and one around the midpoint of the pond. The water samples should be collected from a level midway between the bottom of the pond and the surface of the water.	A/E	As noted above, surface water samples, one near the influent, one near the midpoint, and one near the effluent of the pond will be collected and analyzed for the Contaminants of Potential Concern (COPCs) noted in the UFP-QAPP and summarized on Page 2 of 9 of these RTCs. In the 2011 FFA between the USAF and the USEPA, CS-C503 was identified as a removal action site under Section 6.7.4 and under 6.7.4.2 it is stated that PCBs were discovered in the excavated sediment, which were removed and disposed off-site. The source of the PCBs and the extent of PCB contamination remaining, if any, are unknown. Therefore, the scope of the	

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Comment #	Page	Section/ Paragraph/ Line No.	Comment	A, D, E, FD or X[1]	Response	A or D[2]
					Phase I Remedial Investigation (RI) is to investigate the source and extent of PCB-contamination prior to initiating a removal action, if necessary. The COPCs at CS-C503 were previously determined based on the FFA, Tier I Partnering Meeting discussions, Tier I Meeting minutes and historical sampling results from the maintenance project conducted in 2007 at the Retention Pond. Therefore, adding additional COPCs at CS-C503 is not warranted at this time. It also should be noted that JBA has a stormwater monitoring and maintenance program in place. The JBA stormwater retention ponds are designed and constructed to capture and retain stormwater runoff for the purpose of removing pollutants (including sediment and chemicals), thereby mitigating downstream water quantity impacts. As stormwater flows over land and impervious surfaces, pollutants and sediment are transported downstream into retention ponds designed to allow pollutants and sediment to settle out of solution. Pond plants, algae, and other biological processes remove dissolved metals and nutrients, while natural sedimentation processes remove particulates, organic matter, metals, and other potential pollutants. Some pollutants will not breakdown but will accumulate within pond sediments by design and are removed during maintenance activities such as mucking or dredging. The sediment removed during maintenance activities requires waste profiling to determine disposal criteria in	

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					accordance with the end facility receiving the waste. The stormwater drainage/capture area at CS-C503 consists of parking and driveway areas, commercial buildings, and landscaped and grass areas. In consideration of the CS-C503 land use, it can be reasonably expected that VOCs, SVOCs, PAHs, metals, and pesticides may be present within the CS-C503 Retention Pond sediment by design. These potentially contaminated sediments would be removed and disposed of during regular mucking/dredging maintenance activities as evidenced by the 2007 pond maintenance activities. The 2007 results were consistent with the surrounding land use with the exception of the PCBs detected; therefore, the primary COPC for CS-C503 was determined to be PCBs. DRO and GRO were added as PCBs and are commonly present in petroleum-based oils. In addition, if PCBs are detected above the screening criteria in the soil/sediment samples collected, one sample with the highest concentration of PCBs will be analyzed for dioxins/furans. To ensure that contaminants (including sediment and chemicals) are being retained in the pond as designed, the current JBA Stormwater Monitoring Program samples and analyzes the stormwater outflow immediately downstream of the CS-C503 Retention Pond. The stormwater monitoring program includes the following analyses:	

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					• VOCs by Method 624; • SVOCs by Method 625; • Cyanide by Method 335.4; • Pesticide/PCB by Method 608; • Total Metals by Method 200.7; • Total Nitrate/Nitrite by Method 353.2; • Nitrogen, Kjeldahl by Method 351.2; • Ammonia by Method 350.1; • Phosphorus by Method 365.1; • Chemical Oxygen Demand (COD) by Method 410.4; • Oil/Grease by Method 1664A; • Propylene Glycol by Method SW 8015C; • Total Dissolved Solids (TDS) by Method SM 2540C; • Total Suspended Solids by Method SM 2540D; and • pH. The most recent stormwater monitoring at the CS-C503 outfall occurred on September 18, 2012; the only detections above the reporting limits were: • Ammonia; • Nitrate; and • TDS. The following were detected at low estimated concentrations (between the Method Detection Limit and Reporting Limit): • Bis(2-ethylhexyl) phthalate (common lab contaminant); • Copper; • Zinc;	

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					• Nitrogen, Kjeldahl; • Total Phosphorus; • Chemical Oxygen Demand; and • Total Suspended Solids.	
3		General	It is recommended that soil samples be collected and analyzed from the northwest, north and northeast front of building 1870 (electrical substation). Temporary monitoring wells at the soil collection points might be necessary if the soil samples are positive for the constituents of concern. It is also recommended that building 1870's storm water system be sampled and analyzed for the full suite of analytes.	E	See response to Comment #1d.	
4		General	The laboratory performing the analyses should be required to report Tentatively Identified Compounds (TICs) for all samples analyzed. TIC reporting would aid in the identification of contaminants not previously screened for.	E	TICs are not applicable for the COPCs as defined.	
5		General	If it is determined through sampling and analysis, that contamination is migrating from areas where buildings are present, indoor air inside each building should be sampled and analyzed for the full suite of air toxic constituents.	E	Comment noted. This could be a future consideration if contamination is detected during the Phase I RI.	
6		General	The reviewer understands water samples collected from the CS-C503 Retention Pond Storm Water System may not be available during the time the sampling event is scheduled to occur. If this situation arises, only sediment samples should be collected.	E	Comment noted.	

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7		General	While PCBs have an affinity for soil, water is what provides the transport mechanism for the contaminant. Excluding water samples where recommended above would only prolong the identification of the source of contamination.	E	Comment noted. See response to Comment #1a.	
USEPA Toxicologist (Dawn Ioven) – Comments Received: 1 October 2012 (Comments compiled by Andrew Sochanski, USEPA RPM)						
8	17-1	Wksht #17	Page 17-1, Worksheet#17. Worksheet #17 indicates that the matrices of soil vapor and indoor air are not a concern because "there are no buildings at the site and therefore no potential exposure via the VI pathway." Under *current* site conditions, this is a true statement. However, if VOCs are detected in the subsurface (soil or groundwater) at elevated concentrations, the VI migration pathway *could* be a concern under *future* land- use scenarios and therefore will need to be evaluated.	E	Comment noted. This may be a future consideration if contamination is detected during the Phase I RI.	
USEPA-BTAG (Bruce Pluta and Simeon Hahn) – Comments Received: 1 October 2012 (Comments compiled by Andrew Sochanski, USEPA RPM)						
9		General	The document should provide further clarification describing how the potential COPCs were determined.	E	See the additional summary information on Pages 1 and 2 of 12 of these RTCs.	
10	10-2	Wksht #10	On Page 10-2 it is reported that two composite samples were collected from the sediment in the retention pond for waste characterization and analyzed for VOCs, SVOCs, PCB/pesticides, metals, mercury, TPH, cyanide, sulfides, pH and ignitability. Composite sampling is not recommended for initial waste characterization; therefore, it is recommended that a subset of the	E	The waste characterization sampling was performed in 2007 under a different contract in accordance with the landfill disposal facility permit requirements. The 2007 results were evaluated for potential COPCs. As discussed in Bay West's response to USEPA CRL Comment #2, the only compounds detected	

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			discrete samples being analyzed for GRO/DRO and PCBs be analyzed for the full suite of contaminants. Indicate if the previous composite samples analytical results were compared to screening criteria to determine the COPCs.		that would not be expected in the pond sediments were PCBs. While a comparison was made with the PCB screening levels, the presence of PCBs was sufficient for them to be included as a COPC.	
11		General	The analytical results should be compared to the EPA Region 3 BTAG soil and sediment screening values in addition to the EcoSSLs. Bioaccumulative compounds need to be evaluated for food chain exposures as well. Please see http://www.epa.gov/reg3hwmd/risk/eco/faqs/screenbench.htm	A	The analytical results will be compared to the EPA Region 3 BTAG soil and sediment screening values in addition to the EcoSSLs. Bioaccumulative values and references to those screening values have been added to the Draft Final UFP-QAPP.	
			End Comments			

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Attachment 1

Accident Prevention Plan (on attached CD) and Site Safety and Health Plan

FINAL
PROJECT- WIDE ACCIDENT PREVENTION PLAN

PERFORMANCE-BASED RESTORATION
JOINT BASE ANDREWS NAVAL AIR FACILITY WASHINGTON
CAMP SPRINGS, MARYLAND

Contract W9128F-10-D-0025, DO #0002
OCTOBER 2012
VERSION: 01

Prepared for:

U.S. Air Force
11th CES/CEAN
3466 North Carolina Avenue
Joint Base Andrews, Maryland 20762-4803

US Army Corps of Engineers,
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Prepared by:

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Acronyms and Abbreviations

°C	Degrees Centigrade
°F	Degrees Fahrenheit
µm	micrometer
ACGIH	American Conference of Governmental Industrial Hygienists
AFB	Air Force Base
AHA	Activity Hazard Analysis
AMEC	AMEC Environment & Infrastructure
ANSI	American National Standards Institute
APP	Accident Prevention Plan
ASP	Associate Safety Professional
Bay West	Bay West, Inc.
BBP	Bloodborne Pathogen
bpm	beats per minute
CDC	Center for Disease Control
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CH_4	Methane
CIH	Certified Industrial Hygienist
CNS	Central Nervous System
CO	Carbon Monoxide
CO_2	Carbon Dioxide
CPR	Cardiopulmonary Resuscitation
DO	Delivery Order
DOT	Department of Transportation
DQCR	Daily Quality Control Report
EIT	Engineer-in-Training
EM 385-1-1	Safety and Health Requirements Manual
EMS	Emergency Medical Services
ENG	Engineering
EPA	Environmental Protection Agency
ERP	Emergency Response Plan
ERS	Environmental Remediation Services
eV	electron Volt
HAZWOPER	Hazardous Waste Operations and Emergency Response
IDLH	Immediately Dangerous to Life or Health
ISEA	Industrial Safety Equipment Association
JBA	Joint Base Andrews Naval Air Facility Washington
kV	kilovolts
LEL	Lower Explosive Limit
MDE	Maryland Department of the Environment
MEC	Munitions and Explosives of Concern
mg/m^3	milligrams per cubic meter
min	minute
mph	miles per hour
MSDS	Material Safety Data Sheet
NCP	National Oil and Hazardous Substances Contingency Plan
NFPA	National Fire Protection Association
NWS	National Weather Service
NOAA	National Oceanic and Atmospheric Administration
O_2	Oxygen
OSHA	Occupational Safety and Health Administration
OSIC	On-Scene-Incident-Commander
PCB	Polychlorinated Biphenyl
PE	Professional Engineer
PEL	Permissible Exposure Limit
PG	Professional Geologist
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
ppm	parts per million
QC	Quality Control
RAM	Real-time Aerosol Monitor
RMSF	Rocky Mountain Spotted Fever
RPP	Radiation Protection Program
SARA	Superfund Amendments and Reauthorization Act
SHM	Safety and Health Manager
SOO	Statement of Objectives
SOP	Standard Operating Procedure
SOW	Statement of Work
SPF	Sun Protection Factor
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan

SVOC ..... Semi-Volatile Organic Compound
SZ ..... Support Zone
TA ..... TestAmerica Laboratories
TBD ..... To-Be-Determined
TLV ..... Threshold Limit Value
TWA ..... Time-weighted Average
UFP-QAPP ..... Uniform Federal Policy Quality Assurance Plan
USACE ..... United States Army Corps of Engineers
USAF ..... United States Air Force
UV ..... Ultraviolet
VOC ..... Volatile Organic Compound
WBGT ..... Wet Bulb Globe Temperature
Weston ..... Weston Solutions, Inc.
yd ..... yard
XRF ..... X-Ray Fluorescence

1.0 SIGNATURE PAGE

The following Bay West Inc. (Bay West), project personnel, with teaming partners AMEC Environment & Infrastructure (AMEC) and Weston Solutions, Inc. (Weston) (collectively referred to as "the team"), have reviewed and have agreed to implement and comply with the requirements of the Accident Prevention Plan (APP), for the duration of site activities.

Reviewed by: _____ _____

Daniel Musser, ASP, EIT — Date
Corporate Safety and Health Manager
Bay West
(651) 291-3457

Reviewed by: _____ _____

Doug Hickey, CIH — Date
Corporate Industrial Hygienist
Bay West
(612) 719-9922

Reviewed by: _____ _____

Shirley McMaster, PE — Date
Project Manager
Bay West
(651) 341-3263

Approved by: _____ _____

Marty Wangensteen, PE, PG — Date
Vice President, Federal Programs
Bay West
(651) 291-3475

2.0 BACKGROUND INFORMATION

This APP was prepared for Environmental Remediation Services (ERS) tasks within Joint Base Andrews Naval Air Facility Washington (JBA), Prince George's County, Maryland. Prime contractor Bay West with teaming partners AMEC and Weston (the team) are performing this work under United States Army Corps of Engineers (USACE) Omaha District ERS Contract W9128F-10-D-0025, Delivery Order (DO) 0002.

This APP establishes safety and health procedures, practices and equipment to protect affected personnel from the potential hazards associated with field activities performed at the site. The APP assigns responsibilities, establishes standard operating procedures (SOPs), and provides for contingencies that may arise during the ERS process. This APP interfaces with each team member's Corporate Safety and Health Program.

2.1 Contractor

Bay West, Inc.
5 Empire Drive
Saint Paul, MN 55103

2.2 Contract Number

Contract W9128F-10-D-0025, DO #0002

2.3 Project Information

2.3.1 Project Name

Performance-Based Restoration at JBA, Maryland.

2.3.2 Project Location

JBA is located in Prince George's County, Maryland, near the community of Camp Springs, Maryland. Washington, D.C. is located approximately five miles northwest of the base. The principal features of the base occupy approximately 4,300 acres and consist of runways, airfield operations, an industrial area, and housing and recreational facilities.

JBA was originally established as the Camp Springs Army Air Field on August 25, 1942. The name was changed to Andrews Air Force Base (AFB) in 1947 when the United States Air Force (USAF) was established as a separate military service. The base has served as headquarters at various times for the Continental Air Command, the Strategic Air Command, the Military Air Transport Service, and the Air Force Systems Command. The current major tenant command is the Andrews Naval Air Facility. The missions of the Andrews Naval Air Facility are flight operations and photographic reconnaissance. In 1992, Andrews AFB became an Air Mobility Command Base. In 2009, the name of the base was officially changed to JBA to more accurately reflect the joint nature of the missions and operations at the base.

Work will consist of environmental construction and engineering activities, as necessary, to meet the performance objectives of the Statement of Objectives (SOO). Remediation is being conducted pursuant to: the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), as amended by the Superfund Amendments and Reauthorization Act (SARA), and National Oil and Hazardous Substances Contingency Plan (NCP) requirements; the Maryland Department of the Environment (MDE) Oil Control Program; and the MDE Federal Facilities Program.

2.3.3 Phases of Work and Hazardous Activities

Refer to **Attachment A** for site-specific phases of work. An Activity Hazard Analysis (AHA) for each project task is also included in **Attachment A**.

3.0 STATEMENT OF SAFETY AND HEALTH POLICY

This Statement of Safety and Health Policy is in recognition of the responsibilities of all teaming partners and the need for management to establish a policy with regard to the prevention of on-the-job injuries. The team's primary goal is to minimize the human suffering by employees resulting from occupational injuries through application of these safety policies and procedures. Injuries have a serious physical and emotional impact on the employees themselves and also have a negative effect on family members and co-workers.

In addition, the team recognizes the deterrent and eroding effect injuries have on potential profit. Insurance costs, combined with the indirect costs of injuries, are a matter of serious concern and it is the team's intention they are reduced. This reduction takes place if the frequency of injuries is kept to a minimum. As it affects the team, elimination of on-the-job injuries is an important management responsibility. This responsibility is assumed and treated in the same manner as our business philosophies relating to services rendered.

Each employee must take a serious interest in prevention of injuries for the team's Safety and Health Programs to be effective. Management provides, in administration of this program, leadership and direction to which supervisory personnel and employees will respond. The team requires that all concerned devote their serious attention toward making this Safety and Health Program an integral part of day-to-day business operations. Always remember that no job is so important, and no service is so urgent, that we cannot take time to perform safely.

All site operations will be performed in accordance with applicable federal, state, and local regulations and procedures, Occupational Safety and Health Administration (OSHA) requirements, client requirements, respective teaming partner's Corporate Safety and Health Programs and this APP. Teaming partner employees and their subcontractors will comply with the requirements of this plan.

4.0 RESPONSIBILITIES AND LINES OF AUTHORITIES

All personnel are responsible for continuous adherence to this APP and all safety and health procedures during the performance of their work. No person may work in a manner that conflicts with the intent of, or the inherent safety and environmental precautions expressed in these procedures. The team will dismiss any person who violates safety procedures. Teaming partner employees are subject to progressive discipline and may be terminated for continued violations. All on-site personnel, including subcontractors, will be trained in accordance with this document. No work shall be performed unless a designated competent person is present on the job site.

4.1 Identification and Accountability of Personnel

4.1.1 Corporate Level

4.1.1.1 Project Manager

The Project Manager (PM) is responsible for overall direction, coordination, and successful completion of project field operations. PM health and safety responsibilities include:

- Prepare and organize project activities;
- Review and approve the APP;
- Ensure conformance with corporate and USACE policies and procedures;
- Coordinate project activities with USACE personnel;
- Ensure the project has the necessary resources to operate safely;
- Provide operational/health and safety equipment for project assignments and provide resources and support to the Site Safety and Health Officer (SSHO) for effective completion of duties;
- Ensure project personnel satisfy corporate and USACE Safety and Health requirements;
- Ensure project personnel implement the project APP and established health and safety procedures;
- Ensure project personnel have the appropriate regard for safe job performance;
- Emphasize the importance of safety for effective project management and hold supervision/site personnel and subcontractor management accountable for safe job performance;
- Stop any operation that threatens the health or safety of the team or surrounding population;
- Oversee immediate correction of identified unsafe work conditions/work practices; and
- Monitor and evaluate health and safety performance of project operations.

4.1.1.2 Safety and Health Manager

The Safety and Health Manager (SHM), along with the PM, has authority and responsibility for implementing the APP. The SHM shall have three years of experience managing safety and occupational health at hazardous waste site cleanup operations. The SHM is supported by the Certified Industrial Hygienist (CIH). The SHM responsibilities are listed below:

- Develop the APP before submittal to the USACE for approval;
- Complete revisions or addenda to the APP, as needed;

- Provide for implementation, oversight, and enforcement of the APP;
- Confirm all personnel and subcontractors designated to work on this project are qualified according to medical surveillance and training requirements;
- Be available for emergencies;
- Provide consultation, as needed, to ensure the APP is fully implemented;
- Coordinate any APP modifications with the PM, SSHO, and Contracting Officer;
- Provide continued support for upgrading or downgrading the level of personal protection;
- Evaluate data and recommend changes to engineering controls, work practices, and Personal Protective Equipment (PPE);
- Review accident reports and results of daily inspections;
- Determine and implement personnel disciplinary actions for safety violations;
- Serve as a member of the quality control (QC) staff;
- Stopping any operation that threatens the health or safety of the team or surrounding population;
- Provide additional health and safety, technical assistance to the PM and SSHO; and
- Communicate with the PM to evaluate/resolve health and safety issues.

4.1.1.3 Certified Industrial Hygienist

The CIH is responsible for the oversight in development and coordination of the APP. The CIH is also responsible for the following tasks:

- Modifications to the APP if warranted by changing conditions;
- Determining the level of PPE required;
- Investigation of significant accidents and illnesses and implementation of corrective action plans;
- Overview of monitoring parameters based on expected contaminants;
- Oversight of employee exposure monitoring programs;
- Development of site-specific project Emergency Response Plan (ERP) based on expected hazards; and
- Stopping any operation that threatens the health or safety of the team or surrounding population.

4.1.2 Project Level

4.1.2.1 Field Operations Lead

All site activities will be conducted under the supervision of the Field Operations Lead. The Field Operations Lead oversees normal and emergency work and will perform emergency notifications. The Field Operations Lead is responsible for:

- Supervising all site activities;
- Implementing the field APP;
- Coordinating with the SSHO on safety-related matters;
- Stopping any operation that threatens the health or safety of the team or surrounding population;
- Maintaining logs and records in the field; and

- Implementing changes to APP as directed by the CIH and/or SSHO.

4.1.2.2 Site Safety and Health Officer

Site activities are conducted under the supervision of the SSHO. The SSHO acts as safety oversight for normal and emergency work and will perform any emergency notification as the On-Scene-Incident-Commander (OSIC). The SSHO is responsible for:

- Implementing the field APP;
- Enforcing all provisions of the APP;
- Determining evacuation routes;
- Presenting daily safety meetings;
- Presenting training requirements for site personnel and visitors;
- Maintaining safety logs and records in the field;
- Implementing changes to APP as directed by the CIH and/or SHM;
- General health and safety program administration and enforcement;
- Enforcing the level of personnel protection required;
- Investigating work related accidents and illnesses and implementing corrective action plans;
- Establishing monitoring parameters based on expected contaminants;
- Establishing employee exposure monitoring notification programs;
- Stopping any operation that threatens the health or safety of the team or surrounding population; and
- Upgrading levels of protection based on on-site observations or monitoring results.

4.1.2.3 Work Crew

The work crew has the following responsibilities:

- Immediately report any unsafe or potentially hazardous conditions to the Field Operations Lead, SSHO or SHM;
- Report all incidents, accidents, and near misses, no matter how minor they may seem, immediately to the SSHO;
- Stopping any operation that threatens the health or safety of the team or surrounding population;
- Maintain knowledge of the information, instructions, and emergency response procedures contained in this APP; and
- Comply with the requirements and procedures set forth in this APP, and with any addendums that are added.

4.2 Lines of Authority

Refer to **Attachment A**, Site Safety and Health Plan.

4.3 Stop Work Authority

The PM, SHM, CIH, Field Operations Lead, SSHO, and all on-site workers have authority to stop site activities if any imminently dangerous situation exists. The emergency situation will be immediately reviewed with project team members. USACE also has the responsibility to stop

work immediately if the work is considered a serious threat to the health or safety of workers, other personnel, or to the environment.

When work is stopped due to a hazard or threat to worker health or safety or the environment, the situation and resolution must be documented and submitted to USACE. A pre-task safety and health analysis will be performed for each hazardous task. No work will be performed unless a competent person is on the jobsite. Work must be stopped if unanticipated chemical or biological warfare agents or radiological materials are encountered.

5.0 SUBCONTRACTORS AND SUPPLIERS

5.1 Identification of Subcontractors and Suppliers

Subcontractor personnel associated with this project must adhere to the guidelines and provisions contained in this APP when performing activities at the project Site. The field performance of all subcontractors will be monitored at all times by the Field Operations Lead and SSHO, who will record observations of progress in a formal daily log and discuss project status daily with the PM. The anticipated site-specific on-site contractors include teaming partners Weston and AMEC.

TestAmerica Laboratories (TA) will provide off-site analytical work. Other subcontractors and suppliers on this project are yet to-be-determined (TBD). Prior to project initiation, the team will negotiate and prepare a subcontract agreement to detail the Statement of Work (SOW) and necessary terms and conditions, including safety requirements. Once executed, the team will perform periodic QC reviews.

5.2 Safety Responsibilities of Subcontractors and Suppliers

Subcontractors will provide selected services during performance of the project. Subcontractors who come on-site to perform field site work or enter controlled areas of the site are subject to APP requirements. Subcontractor health and safety responsibilities include:

- Provide necessary health and safety documentation to the SSHO as indicated below (as applicable):
  - Hazardous Waste Operations and Emergency Response (HAZWOPER) training (40-hour worker, 8-hour supervisor, 8-hour refresher);
  - First aid/ Cardiopulmonary Resuscitation (CPR) training;
  - Other health and safety training as appropriate (e.g. competent person, permit-required confined space);
  - Medical clearance (fitness for duty) certification; and
  - Respirator fit testing.
- Provide a hazardous substances inventory list and copies of applicable material safety data sheets (MSDSs) to the SSHO for hazardous substances brought on site by the subcontractor;
- Subcontractor supervisors are required to enforce compliance with applicable APP requirements by their employees;
- Review, understand, and comply with the APP and safety instructions from the SHM, SSHO, PM, or other competent authority;
- Promptly report all injuries or illnesses to their supervisor and the SSHO; and
- Immediately report any unsafe work conditions, unsafe work practices, and violations of the APP to their supervisor and the SSHO.

All personnel will acknowledge they have read, understood and will abide by the APP for this project, by their signature. At all times, subcontractors shall abide by the guidance given by the Field Operations Lead or SSHO. Any deviations from the site plans can be used as the basis for termination of the subcontract agreement.

6.0 TRAINING

6.1 Safety Indoctrination

The following subjects are included in the project safety indoctrination:

- SOO;
- General safety and health policy and procedures;
- Decontamination procedures;
- ERP, including the necessary PPE and other equipment;
- Employee and supervisor responsibilities for reporting all accidents;
- Excavation and Trench Safety;
- Hearing Conservation;
- Fire Protection and Prevention;
- Hand and power tool safety;
- Hazard communication;
- Job hazards and the means to control/eliminate those hazards, including applicable position and/or activity hazard analyses;
- Medical surveillance requirements, including recognition of symptoms and signs indicative of overexposure to hazards;
- Names of personnel and alternates responsible for site safety and health;
- Procedures for reporting and correcting unsafe conditions or practices;
- Provisions for medical facilities and emergency response and procedures for obtaining medical treatment or emergency assistance;
- Requirements and responsibilities for accident prevention and maintaining safe and healthful work environments;
- Respiratory protection;
- Safe use of engineering controls and equipment on the site;
- Safety, health, and other hazards present on-site;
- Spill containment;
- Use of PPE;
- Work practices by which the employee can minimize risks from hazards; and
- Safe lifting techniques.

All site personnel must receive training and acknowledge understanding of the contents of this APP prior to performing work at the site. This training is documented on the initial Training Acknowledgement Form (**Appendix 1**).

6.2 Mandatory Training and Certification

All project field personnel have received the training and or certifications listed below:

- 40-hour HAZWOPER training in accordance with 29 Code of Federal Regulations (CFR) 1910.120 (e);
- 8-hour annual refresher training in accordance with 29 CFR 1910.120 (e);
- 3 days of field experience under the direct supervision of a trained experienced supervisor in accordance with 29 CFR 1910.120 (e);

- First Aid / CPR / Bloodborne Pathogen (BBP) training [minimum of two personnel on-site];
- 8-hour Supervisory Personnel Training (Field Operations Lead) in accordance with 29 CFR 1910.120 (e);
- All heavy equipment operators will be skilled and knowledgeable at the level of competent person, as defined by OSHA for all excavation activities; and
- Annual physicals with medical clearance in accordance with 29 CFR 1910.120 (f).

All current certifications and training certificates for site personnel will be maintained on-site for the duration of the project. Individuals without proper training records are not permitted on-site.

6.3 Emergency Response Training

All project personnel will receive the following emergency response training at the beginning of the project and will also receive periodic refresher training:

- Fire extinguisher use;
- Spill response;
- Severe weather; and
- Medical emergency.

6.4 Safety Meetings

The SSHO will conduct a daily safety briefing that covers the day's planned operations, reviews the previous day's safety considerations and reviews new safety issues encountered. If no new safety issues are encountered, at least one relevant safety item will be briefed.

In addition to daily safety briefings, the SSHO will conduct weekly training. The training shall address safety and health procedures, work practices, changes in the APP or AHA, work tasks, or schedule. The SSHO will conduct site-specific training sessions for new personnel, visitors, and suppliers using information supplied in this APP.

Safety meetings and training shall be documented on the Safety and Health Meeting Report Form (**Appendix 2**). This form shall be completed and signed by each person attending training. General topics will be listed on the form. The SSHO maintains copies of all documentation.

The leader of each field team shall conduct a tailgate safety brief at the work site to discuss local conditions and hazards at the beginning of each shift or when beginning work at a new site. Topics included in the briefing include:

- PPE;
- Medical Surveillance;
- Exposure Monitoring/Air Monitoring;
- Heat and Cold Stress Management;
- SOPs, Engineering Controls and Work Practices;
- Site Control Measures;
- Personal Hygiene and Decontamination;
- Equipment Decontamination;
- Emergency Equipment and First Aid;
- BBP;
- Emergency Response and Contingency Procedures; and
- Logs, Reports and Recordkeeping.

7.0 SAFETY AND HEALTH INSPECTIONS

General safety and health inspections are described throughout this APP. The SSHO will conduct safety inspections on a daily basis or more frequently if conditions warrant.

The SSHO is responsible for daily safety inspections of the project. A safety inspection checklist form will be used to record, track and provide follow-up to ensure safety deficiencies are corrected after they are identified. A record of the safety inspection will be maintained in the project file. Deficiencies will be identified, posted, and dated when the deficiencies are rectified. Deficiencies and corrective actions will be communicated to USACE via the Monthly Safety Exposure Report.

7.1 Internal Inspections

The SSHO is responsible for daily inspections of the project. The SHM and/or CIH may make additional random inspections as warranted. The areas to be inspected include, but are not limited to:

- Fire extinguishers;
- First Aid Kits;
- Vehicles;
- Observance of health and safety precautions; and
- PPE.

7.2 External Inspections

No external inspections are anticipated; however, a regulatory agency may conduct a site inspection at any time. Personnel who cannot demonstrate their affiliation with a recognized regulatory agency should not be allowed access to the project site.

8.0 ACCIDENT REPORTING

8.1 Exposure Data

The Field Operations Lead will report hours worked in the Daily Quality Control Report (DQCR). The PM will prepare Monthly Safety Exposure Reports. The report will encompass on-site work by all hourly and salaried employees as well as all of the subcontractors working on this project. A copy of the exposure report is sent to the Contracting Officer no later than the fifth work day of each month.

8.2 Accident Investigations, Reports, and Logs

All accidents, including "near incidents" or "near misses" are investigated. The lead accident investigator for the project is the SSHO. The SSHO will log each such incident, recording the dates and times when the event occurred, when the event was reported, when the response began, and when the investigation was completed, as well as documenting the investigation procedures and outcome (**Appendix 5**).

This section provides the requirements for implementing the accident reporting provisions of the USACE Safety and Health Requirements Manual (EM 385-1-1). This APP requirement applies to all work performed on the project.

The PM and the SHM and/or CIH will be notified immediately by telephone of any accidents and follow-up with USACE's Accident Investigation Report form. All accidents are investigated internally.

Person(s) who become ill or injured during work activities will immediately inform their direct supervisor, regardless of the severity of the illness or injury. If the injury occurred in contaminated areas the victim(s) will be decontaminated. In the event the medical emergency is severe enough, the SSHO will order a cessation of work and notify off-site emergency personnel. All personnel at the work site shall use the buddy system, staying within sight of their partner. If a partner becomes incapacitated or severely ill, an ambulance will be called. In the event that a cessation of work is ordered, all personnel should:

- Assist the SSHO and/or supervisor, if required, in decontaminating the victim and/or administering first aid;
- Leave the contaminated area and undergo decontamination prior to entering the worker rest area; and
- Assist emergency response personnel when requested.

In the event of an accident that results in a lost workday or $2,000 or more in property damage, an Engineering (ENG) Form 3394 (**Appendix 4**) is completed and submitted within five workdays to USACE.

All workers receiving medical treatment by a physician will obtain a release from the physician on the date of treatment stating one of the following: (1) the employee is not fit for duty; (2) the employee is fit for restricted duty; or (3) the employee is fit for duty. A copy of the release will be attached to the accident report (ENG Form 3394) and submitted to USACE.

In addition to USACE reporting requirements, all injuries, accidents and illnesses will be reported and recorded in accordance with OSHA reporting requirements.

8.3 Major Accident Notifications

Should an accident occur resulting in a fatality, permanent or partial disability, $200,000 or more in property damage, three or more persons being hospitalized, or possible adverse publicity to

USACE, immediate notification will be made to the USACE PM in person, telephonically, or by email. The reporting requirement of submitting ENG Form 3394 within five working days applies. If a subcontractor employee is affected the SSHO will also contact the respective safety and health contact for the subcontractor. The SHM will make notifications to OSHA when appropriate.

9.0 MEDICAL SUPPORT

In a true emergency, apply first aid and call **911**. Be prepared to brief the operator on all details such as location of the emergency and the nature and extent of injuries. Preservation of life and limb is the priority. Some discretion is required as to whether to call Emergency Medical Services (EMS) or transport or seek urgent versus emergency care; when in doubt, **call 911**.

9.1 On-Site Medical Support

Selected employees are trained in the first aid treatment skills and CPR. The first aid course includes BBP training and prevention. These trained employees will have their current certificate cards on-site. A minimum of two employees will be certified in CPR and first aid per site. First aid kits will be provided in each project vehicle and at the site office.

9.2 Off-Site Medical Support

All emergency medical treatment, other than first aid, will be coordinated by EMS. **Attachment A** lists site emergency telephone numbers. All first aid will be administered on-site by the SSHO or designee, who is certified in CPR and first aid. There will always be at least two individuals trained in first aid and CPR available during site activities. First-aid supplies will be kept in the support zone (SZ). All vehicles used to transport injured persons to the medical facility will be provided with directions and a map to the medical facility.

The closest hospital to Site, directions, mileage, drive time, and map are included in **Attachment A**.

9.3 Non-Emergency Treatment

A non-emergency injury or illness that may require treatment beyond on-site first aid may be treated at the following clinic:

Concentra Medical Center
9141 Alaking Court Suite 112
Capitol Heights, MD 20743
(301) 499-4655

9.4 Medical Surveillance

9.4.1 Medical Examination

All personnel performing work that may result in exposure to contaminant-related health and safety hazards shall be enrolled in a medical surveillance program. They will have successfully completed a pre-placement occupational physical examination and annually thereafter. Certification of Medical Clearance is also appended for review.

This examination is designed to meet the requirements of 29 CFR 1910.120 (f) requirements for hazardous waste site operations.

The medical surveillance provided to the employees includes a judgment by the medical examiner of the ability of the employee to use either positive or negative pressure respiratory equipment in accordance with 29 CFR 1910.134. Any employee found to have a medical condition that could directly or indirectly be aggravated by exposure to chemical substances or by the use of respiratory equipment will not be employed for projects requiring clearance under the Respiratory Protection Program. A copy of the medical examination is provided at the employee's request.

The employee is informed of any medical conditions that result in work restriction or that would prevent them from working at hazardous waste sites.

Contractors will certify that all their employees have successfully completed a physical examination by a qualified occupational health physician and will supply certification of medical clearance for each on-site employee.

9.4.2 Medical Restriction

If an occupational injury or illness occurs that restricts an employee's ability to function at full capacity, the team maintains a policy of providing these employees with restricted duty assignments whenever possible to allow them to continue to be productive.

9.4.3 Medical Records

Medical and personal exposure monitoring records will be maintained according to the requirements of 29 CFR 1910.120 (f) and shall kept for a minimum of 30 years. Employee confidentiality is maintained.

10.0 PERSONAL PROTECTIVE EQUIPMENT

PPE for this project was carefully selected after due consideration of the hazards involved. During field work, site conditions will be closely monitored for changes in conditions or the presence of unanticipated hazards. If necessary, the SSHO will adjust PPE levels. The SHM and/or CIH may assist in the decision to adjust PPE levels.

The SSHO will train all site personnel on PPE before beginning work. Site personnel must demonstrate understanding of the training and the ability to correctly use the PPE. Completion of PPE training is documented on the Training Acknowledgement Form (**Appendix 1**).

10.1 PPE Selection Decision Making Criteria

PPE will be worn to minimize personnel exposure to site contaminants. Decision-making criteria for PPE selection requirements include:

- Site historical information and work location;
- Type of exposure potential suspected or known to be present (i.e. chemical hazards, physical agents, physical hazards, biological hazards, etc.);
- Duration of site activities;
- Type of task to be performed; and
- OSHA and team requirements.

The SSHO will enforce the level of protection worn by site personnel. Levels of protection will be upgraded or downgraded at the discretion of the SSHO based on real-time air monitoring data and site experience. Any changes in the level of protection will be documented. The requirements outlined below are based upon the substances present, potential for contact, and chemical resistance of protective clothing, in accordance with current information.

10.2 Proper PPE Use

All site personnel will be given site-specific PPE training as part of the site-specific safety training required by OSHA 29 CFR 1910.120(g). Personnel will receive training on each item of PPE they will be required to use. Minimum requirements will include the need for PPE, proper use, proper donning/removing, and PPE limitations. Donning/removing procedures will be demonstrated during the initial site-specific safety training.

PPE training will be conducted by the SSHO prior to the site worker using a specific PPE item. All site personnel will be properly fitted for each item of PPE required for this project. PPE will be used in accordance with OSHA 29 CFR 1910 Subpart I. PPE decontamination will be performed in accordance with **Attachment A**, Site Safety and Health Plan.

10.3 PPE Hazard Assessment

Chemical degradation or permeation of PPE and worker heat/cold stress can significantly affect the length of time a person can work in PPE. Based on the chemical contaminants of concern concentrations and the anticipated ambient air temperatures during this project, limitations on the length of time a person can work outside in the heat/cold may be expected. Site workers will be monitored for potential heat stress illnesses and cold stress injuries.

Site workers are encouraged to report any perceived problem or difficulties with PPE to the SSHO, including any signs or symptoms of heat stress (e.g. rapid pulse, nausea, or fatigue), any signs or symptoms of cold stress (e.g. sluggish pulse, blanching or whitening of the skin, shivering, numbness, or drowsiness), any problems/difficulties such as chest pain, discomfort

interference with vision or communication, restriction of movement, unusual residues on PPE, or skin irritation.

10.4 Maintenance and Inspection of PPE

PPE will be maintained and stored in accordance with the manufacturers' recommendations. PPE at this project will be stored in the SZ. The SSHO will be responsible for maintaining, storing, and issuing project PPE. Once an item of PPE is issued to a site worker, its maintenance, inspection, and storage becomes the responsibility of the individual site worker. PPE will be inspected by the SSHO prior to issuing it, and by the site worker receiving the PPE, in accordance with the manufacturers' recommendations. Site worker inspections will also be conducted before each use.

10.5 General PPE Requirements

Clothing – Site workers must wear long pants and shirts with sleeves, or coveralls. T-shirts may be worn at the discretion of the SSHO as conditions warrant. No loose clothing, hooded attire, dangling jewelry, loose long hair, or rings are permitted around heavy equipment, drill rigs, or rotating machinery.

Foot protection – Safety toe work boots with ankle support. Types of safety shoes may vary by specific job requirements. Open shoes are strictly forbidden on job sites. Rubber safety shoes or rubber boots over safety shoes will be worn if site conditions are wet/muddy/slippery.

Head protection – All personnel are required to wear head protection when working around overhead hazards.

Hearing protection – Disposable earplugs and earmuffs will be provided to all employees potentially exposed to excessive or chronic noise levels.

Eye/face protection – Employees must use eye and face protection when machines or operations present potential eye or face injury from physical or chemical agents. All eye and face protection must meet Industrial Safety Equipment Association (ISEA)/American National Standards Institute (ANSI) standard Z87.1. Regular prescription eyewear or sunglasses are not acceptable for protection against impact unless made with polycarbonate safety lenses and frames. Safety glasses with side shields will be worn at all times on-site.

10.6 Levels of Protection

Refer to **Attachment A**, Site Safety and Health Plan.

10.7 Respiratory Protection

Where a potential respiratory hazard exists, respiratory protection will be supplied to affected employees. Only approved respiratory protective equipment that has been specifically selected for the project will be used. Employees will be instructed on proper fit, maintenance procedures of equipment, and warning signs of respiratory equipment failure.

The use of all respiratory protective equipment will conform to the manufacturer's operating instructions and training provided to the employee. In all environments where it is determined respiratory protection is necessary, ambient air monitoring will be required. All personnel on-site must be medically cleared and fit-tested for each type of respiratory protection they will use.

11.0 PLANS, PROGRAMS, AND PROCEDURES REQUIREMENTS

The following plans, programs, and/or procedures are required by USACE EM 385-1-1.

11.1 Layout Plans

Not applicable.

11.2 Emergency Response Plans

11.2.1 General

This section provides information regarding the action(s) to be taken in the event an emergency situation develops. In the event of an emergency, the PM, Field Operations Lead, and SSHO are authorized to stop work. The SSHO will implement the ERP whenever conditions at the site warrant an emergency response.

11.2.2 Pre-Emergency Planning

Decisive action is required if an emergency occurs. Decisions must be made immediately and personnel must be ready to immediately respond to an emergency. For this reason, pre-emergency planning is an essential part of each project's ERP. Pre-emergency planning tasks will be developed and established prior to the start of site work. Pre-emergency planning for the project includes the following tasks:

- Development and approval of this ERP section;
- Coordination of the ERP with emergency response personnel;
- Training site personnel in emergency procedures;
- Maintaining emergency response equipment on-site;
- Conducting an emergency response practice drill before site activities begin; and
- Modification of the ERP, if necessary, as work progresses.

11.2.3 Response Priorities and Procedures

The following outline provides guidance in prioritizing emergency response action and provides general response procedures to be followed. On-site personnel will only provide minimal or first line response to all emergencies. In the event of an emergency, call **911** from a base phone or (301) 981-9911 from a cell phone that is the primary point of contact (POC), and the SSHO will coordinate the appropriate response.

First Priority: Prevent further injury or illness by:

- Protecting response personnel;
- Isolating the scene to authorized personnel only;
- Notifying emergency response personnel; and
- If possible, rescuing any injured parties.

Second Priority: Provide first aid to persons with life-threatening injuries or illnesses.

Third Priority: Alleviate the immediate hazards by:

- Extinguishing incipient stage fire;
- Reducing chemical releases; or
- Containing any spill.

11.2.4 Evacuation Routes and Procedures

In a severe emergency (e.g. a large fire, explosion, or large chemical release) site evacuation may be necessary. The SSHO is responsible for informing site personnel of the evacuation routes for each worksite during the morning safety briefing. The evacuation route and assembly area will correlate to the wind direction, topography, and the nature of the incident.

Personnel will be advised to move to an upwind location at least 100 yards (yds) from any fires and/or releases, and are advised to continually monitor wind direction for changes. If moving upwind is not possible without encountering the incident, personnel will be advised to move cross wind or downwind a distance necessary to be out of the path of vapor releases, smoke, odors, or spills. In the event that a site evacuation becomes necessary, the following procedures (see **Table 11-1**) will be used:

Table 11-1 Site Evacuation Procedures

Step	Task
1.	Site personnel are notified of an emergency evacuation via portable hand-held radios, an air horn signal, or verbal command. All site personnel will immediately stop work.
2.	All site personnel evacuate the work area as quickly as possible, and assemble at a site-specific location at least 100 yds upwind of the incident, or as instructed during the morning safety briefing.
3.	The SSHO is responsible for initiating a roll call.
4.	The SSHO will contact EMS as all site personnel are being accounted for during roll call.
5.	The SSHO will ensure EMS has adequate site access.
6.	The SSHO will ensure all combustion equipment has been shut down.
7.	All site personnel assembled at the designated safe evacuation area will wait for further instructions from emergency response personnel.

11.2.5 Community Notification

The major hazards posed to employees and visitors on the project site are from chemical and physical hazards from site activities. Potential chemical and physical hazards are described in **Section 11.7**. Establishing and implementing site control measures mitigates hazards to protect employees and the public.

If site activities require road closure, restricting access to an area by employees or visitors, or if an emergency situation requires the evacuation of nearby employees and visitors, the SSHO will coordinate these events with the installation POC. After community notification procedures have been started, the SSHO will notify the PM, who will contact the USACE.

11.2.6 Emergency Medical Treatment and First Aid

Refer to **Section 9.0**, Medical Support and **Attachment A**.

11.2.7 Emergency and First Aid Equipment

A complete first aid kit, Type III, 16 unit or larger in a waterproof container, and containing at a minimum, a one-pocket mouthpiece for CPR, absorbent compresses, adhesive bandages, adhesive tape, antiseptic swabs, burn gel, sterile pads, and a triangular bandage will be will be located in the SZ. Its contents will be evaluated and possibly modified for this specific project. The contents will be checked prior to their utilization for sterility and to replace expended items. The SSHO or another designated individual will inventory the kit weekly.

Prior to the start of work, the SSHO will discuss with site personnel the prevention steps, symptoms and medical persons available to assist with injuries or questions on diseases, plants or animals that could be encountered while working on this project.

A working cell phone and radio with adequate signal in this area will be maintained on-site and fully charged at the start of each work day.

A fire extinguisher, (dry chemical or carbon dioxide with a minimum rating of 4-A:60-B:C) will be readily available located in the SZ. A fire extinguisher with a minimum rating of 2-A:10-B:C will be maintained in each vehicle as well. Personnel will be instructed on the proper use of fire extinguishers.

11.2.8 Discovery of a Questionable Item

Work must be stopped if munitions and explosives of concern (MEC) items, biological warfare agents or radiological materials are encountered. Evacuate a safe distance in the upwind direction and contact the PM.

11.2.9 Decontamination during a Medical Emergency

For minor medical problems or injuries, regular decontamination procedures will be followed. If emergency, life-saving first aid and/or medical treatment are required, regular decontamination procedures may need to be abbreviated or omitted. The SSHO will advise EMS and hospital medical staff of the type of contamination.

- Do not attempt to wash or rinse the individual, unless the individual has been contaminated with an extremely toxic or corrosive chemical that may cause injury or loss of life to emergency response personnel;
- Outer garments can be removed if it does not cause a delay, interfere with treatment, or aggravate the problem;
- PPE can be cut away, and respiratory protective equipment must always be removed; and
- If contaminated clothing cannot be safely removed, then the individual should be wrapped in a blanket or plastic sheeting to prevent the contamination of the inside of the ambulance and/or emergency response personnel.

11.2.10 Hazardous Substance Spill or Release

Hazardous substance spill or release situations may be different due to the way the incident occurred, how hazardous the substance is, and how much was spilled or released. If a hazardous substance spill or release occurs, the following steps will be taken:

- Evacuate site personnel if necessary;
- Determine the source of leak or release;
- Determine the approximate volume of the leaked or released substance and identify the contaminants involved;
- Contact **911** to inform them of the spill or release;
- Don appropriate PPE;
- Secure the spread of the spill, if possible, using one of the following methods of containment:
    - Patch and plug;
    - Sorbent materials (e.g. clay, saw dust, absorbent pillows, sheets, or rolls);

- Dikes; and/or
- Damming with soil straw bales, or sand bags.

- After emergency response personnel have been contacted **through 911**, notify the PM, SHM, and the USACE POC.

11.2.11 Chemical Overexposure

In case of accidental overexposure to a hazardous chemical/material, the following guidelines (**Table 11-2**) will be used:

Table 11-2 Chemical Exposure First Aid Guidelines

Type of Exposure	First Aid Guideline
Skin Contact	Skin: Wash/rinse the affected area thoroughly with copious amounts of soap and water.
	Eyes: Rinse eyes for at least 15 minutes following chemical contamination.
	Contact EMS, if required, or transport individual to the hospital. Move the individual to fresh air.
Inhalation	Contact EMS, if required, or transport individual to the hospital. Contact Poison Control Center.
Ingestion	Contact EMS or transport individual to the hospital.

11.2.12 Heat Stress Illness / Cold Stress Injury First Aid

11.2.12.1 Heat Rash

Heat rash is caused by continuous exposure to heat and humid air and is aggravated by wet chafing clothing. This condition decreases a worker's ability to tolerate hot environments.

- **Symptoms**: Mild red rash, especially in areas of the body that sweat heavily.
- **Treatment:** Decrease amount of time in protective gear and provide powder such as cornstarch or baby powder to help absorb moisture and decrease chafing. Maintain good personal hygiene standards and change into dry clothes if needed.

11.2.12.2 Heat Cramps

Heat cramps are caused by a profuse rate of perspiration that is not balanced by adequate fluid and electrolyte intake. The occurrence of heat-related cramps is an indication that excessive water and electrolyte loss has occurred, which can further develop into heat exhaustion or heat stroke.

- **Symptoms:** Acute, painful spasms of voluntary muscles such as the back, abdomen and extremities.
- **Treatment:** Remove victim to a cool area and loosen restrictive clothing. Stretch and massage affected muscles to increase blood flow to the area. Have patient drink one to two cups of liquids immediately, and every twenty minutes thereafter. Consult with physician if condition does not improve. If available, an electrolyte replacement solution should be taken along with liquids.

11.2.12.3 Heat Exhaustion

Heat exhaustion occurs due to large fluid and salt loss from profuse sweating. It is a state of very definite weakness or exhaustion caused by increased stress on various organs to meet

increased demands to cool the body due to excessive loss of body fluids. This condition leads to inadequate blood supply and cardiac insufficiency.

Heat exhaustion is less dangerous than heat stroke but nonetheless must be treated. If allowed to go untreated, heat exhaustion can quickly develop into heat stroke.

- **Symptoms:** Pale or flushed, clammy, moist skin, profuse perspiration, and extreme weakness. Body temperature is basically normal or slightly elevated, the pulse is weak and rapid, and breathing is shallow. The individual may have a headache, be dizzy or nauseated.
- **Treatment:** Remove the individual to a cool, air-conditioned place, loosen clothing, elevate feet and allow individual to rest. Consult physician, especially in severe cases. Have patient drink one to two cups of liquids immediately, and every twenty minutes thereafter. Total liquid consumption should be about one to two gallons per day. If the signs and symptoms of heat exhaustion do not subside, or become more severe, immediate medical attention will be required.

11.2.12.4 Heat Stroke

Heat stroke is an acute and dangerous reaction to heat stress caused by failure of the heat regulating mechanisms of the body. Heat stroke occurs when the body's system of temperature regulation fails and the body temperature rises to critical levels. When this occurs, the body core temperature rises very rapidly to a point (>105.8°F) where brain damage and death may result if the person is not cooled quickly.

- **Symptoms:** The victim's skin is hot, and may or may not be red, dry and/or spotted, due to the fact that the individual may still be wet from having sweat while wearing protective clothing earlier; nausea; dizziness; confusion; extremely high body temperature; rapid respiratory and pulse rate; delirium; convulsions; unconsciousness or coma.
- **Treatment:** Cool the victim immediately. If the body temperature is not brought down quickly, permanent brain damage or death may result. The victim should be moved to a shady area; lie down and keep their head elevated. Cool the victim by either sponging or immersing the victim in very cool water to reduce the core temperature to a safe level (<102°F). If conscious, give the victim cool liquids to drink. Do not give the victim caffeinated or alcoholic beverages.
- Observe the victim and obtain immediate medical help. Heat stroke is considered a medical emergency. Medical help should be summoned immediately.
- **EARLY RECOGNITION AND TREATMENT OF HEAT STROKE ARE THE ONLY MEANS OF PREVENTING BRAIN DAMAGE OR DEATH.**

11.2.12.5 Frostbite

Frostbite is both the general and medical term given to areas of cold injury. Unlike hypothermia, frostbite rarely occurs unless environmental temperatures are less than freezing and usually less than 20°F. Frostbite injuries occur most commonly on the distal parts of the body (nose, earlobes, hands, and feet) that are subject to intense vasoconstriction.

- **Symptoms:** The three general categories of frostbite are:
  - Frostnip - A whitened area of the skin, which is slightly burning or painful;
  - Superficial frostbite - Waxy, white skin with a firm sensation but with some resiliency. Symptomatically feels "warm" to the victim With a notable cessation of pain; and

- Deep frostbite - Tissue damage deeper than the skin, at times, down to the bone. The skin is cold, numb and hard.

- **Treatment:** **Frozen tissue** is a **medical emergency**, and the individual must receive medical attention immediately. Contact EMS or transport the individual to the hospital.

11.2.12.6 Hypothermia

Hypothermia is a life-threatening condition if the core body temperature falls below 95°F. Hypothermia can occur at temperatures above freezing, particularly when the skin or clothing becomes wet. The ability to sustain metabolic rate and to reduce skin blood flow is diminished by fatigue. Thus, fatigue increases the risk of severe hypothermia by decreasing metabolic heat. Additionally, because blood flow through the skin is reduced to conserve heat, the skin and underlying tissues become more susceptible to frostbite..

- **Symptoms:** During exposure to cold, maximum shivering occurs when the core temperature falls to 95°F. As hypothermia progresses, depression of the CNS becomes increasingly more severe accounting for the progressive signs and symptoms ranging from sluggishness and slurred speech to disorientation and eventually unconsciousness (**Table 11-3)**.

Table 11-3 Progressive Clinical Symptoms of Hypothermia

Core Temperature (°F)	Clinical Signs
95°	Maximum shivering
87° - 89°	Consciousness clouded; blood pressure becomes difficult to obtain; pupils dilated
84° - 86°	Progressive loss of consciousness; muscular rigidity; respiratory rate decreases
79°	Victim rarely conscious
70° - 72°	Maximum risk of ventricular fibrillation

- **Treatment:**
  - First aid treatment for **mild hypothermia** includes the use of heat to raise the individual's body temperature. Heat may be applied to the individual in the form of heat packs, hot water bottles, and blankets. If the individual has not recovered within half an hour, then transport the individual to the hospital for medical attention.
  - **Severe hypothermia** is a ***medical emergency***, and the individual must receive medical treatment immediately. First aid treatment for severe hypothermia includes handling the individual very gently; rough handling may set off an irregular heartbeat. ***DO NOT*** attempt to re-warm the severely hypothermic individual without professional medical assistance; re-warming may cause the development of an irregular heartbeat. Contact EMS or transport the individual to the hospital.

11.2.13 Snake Bite First Aid

In the event of a snake bite emergency, the SSHO will take the following actions (**Table 11-4**). ***DO NOT*** apply ice, cut the bite wound, or apply a tourniquet to a snake bite.

Table 11-4 First Aid Procedure for a Snake Bite

Step	Task
1.	Try to keep the victim calm.
2.	Contact EMS immediately.
3.	Wash the bite wound.

Table 11-4 First Aid Procedure for a Snake Bite

Step	Task
4.	If the snake bite is on the victim's arm or leg: • Keep the bitten area below the level of the victim's heart. • Splint the appendage to keep it immobile.
5.	If necessary, carry the victim to emergency transportation, or have the victim walk slowly.

11.2.14 Insect Bites/Stings First Aid

Signs of spider bites and scorpion stings are similar to other sudden illnesses. The signs include:

- Nausea or vomiting;
- Difficulty breathing; and/or
- Sweating or salivating more than usual.

A spider bite may also be suspected when there is also severe pain in the bite area, a mark indicating a possible bite, or swelling of an area. In the event of a spider bite emergency, follow the procedures listed in **Table 11-5**:

Table 11-5 First Aid Procedures for a Spider Bite

Step	Task
1.	Wash the wound/swelling area with soap and water.
2.	Cover the wound area to keep it clean.
3.	Apply an ice pack to the wound/swelling to reduce any pain and swelling.
4.	Contact EMS immediately.

In the event a site worker is bitten/stung by an insect, follow the procedures listed in **Table 11-6**:

Table 11-6 First Aid Procedures for an Insect Bite / Sting

Step	Task
1.	Remove the stinger if possible. Scrape the stinger away from the skin with a fingernail, or use tweezers. If tweezers are used, be sure to grasp the stinger and not the venom sac.
2.	Wash the bite/sting area with soap and water.
3.	Cover the bite/sting area to keep it clean.
4.	Apply an ice pack to the bite/sting area to reduce any pain and swelling.
5.	Watch the victim for signals of an allergic reaction.

EMS should be contacted in the event of an insect bite/sting:

- If the individual does not know what bit/stung them;
- If the individual has a history of allergic reactions to insect bites/stings;
- If the individual is bitten/stung on the face or neck; or
- If the individual begins to have difficulty breathing.

In the event a site worker finds a tick attached to their skin, follow the procedures listed in **Table 11-7**:

Table 11-7 First Aid Procedures for Tick Removal

Step	Task
1.	To minimize the chance you will contact a tick-borne illness, remove the tick quickly. Use blunt tweezers, grasp the tick firmly and close to the skin, and then pull up. Avoid squeezing the tick's body.
2.	Wash the bite area with soap and water.
3.	Cover the bite area to keep it clean.
4.	Instruct worker to report to SSHO if they experience redness or swelling at the site of the tick bite.

11.2.15 Adverse Weather Conditions

In the event of adverse weather conditions, the SSHO will determine if work can continue without sacrificing the health and safety of site personnel. The SSHO will monitor threatening weather conditions via radio, television, Internet, or calls to the National Weather Service (NWS). Some of the conditions considered by the SSHO include:

- Potential for heat or cold stress;
- Limited visibility;
- Electrical/thunderstorms;
- Hurricane watch or warning;
- Tornado warning or sighting; and
- Treacherous weather-related working conditions (e.g. heavy rainfall, high winds, icy conditions causing slippery footing hazards).

11.2.16 Site Security and Control

The SSHO is responsible for site security and includes the control of entry or exit of personnel and equipment to the site in an emergency. The SSHO coordinates the arrival of any emergency response personnel. Unauthorized persons will not be permitted to enter the site during an emergency incident.

11.2.17 Emergency Communications

Communications during site emergencies will include the following procedure:

- Contact **911** first; then contact the contact PM, SHM, and USACE POC;
- On-site communications with site personnel using hand-held, portable radios and telephones;
- On-site communications using air horns and verbal messages; and
- Off-site communications using the telephone; **Attachment A** provides a list of emergency telephone numbers.

An air horn or vehicle horn alarm system that has the adequate means of site communication (i.e. convey information above ambient noise levels) will be made available to each work crew. Horns can be used to alert site personnel of an emergency situation. The following signals (**Table 11-8**) will be used:

Table 11-8 Air Horn Signals

Signal	Meaning
One long blast for at least 10 seconds	Immediately evacuate the site and assemble at the safe evacuation location identified by the SSHO at the morning safety briefing.
Three short blasts	There is an emergency on-site that requires first aid assistance.

11.2.18 Procedures and Tests

The SSHO will conduct at least one emergency response drill during the field effort to test emergency response capabilities. Drills are documented on the DQCR and in the SSHO log book.

11.2.19 Spill Plan

In the event of a spill of hazardous material, the SSHO shall assume control and direct efforts for controlling and remediating the incident and reporting the incident to the PM.

The most likely hazardous material release event to occur is the release of fuel or hydraulic fluid. Other hazardous materials are not anticipated for use in significant quantities at the site.

Level D PPE is adequate for controlling and remediating spills and discharges based upon the anticipated types and quantities of materials on-site,

The SSHO shall manage all releases of hazardous materials. Sorbents and spill control materials will be provided on-site for use in the event of a release of hazardous materials. Storage of contaminated material or hazardous materials are to be appropriately bermed, diked and/or contained to prevent any spillage of material on uncontaminated soil. If the spill or discharge is reportable, and/or human health or the environment is threatened, the SSHO will notify the USACE POC.

11.2.20 Firefighting Plan

11.2.20.1 Small / Incipient Fire

A small fire is defined as a fire that can be extinguished with a fire extinguisher. An incipient fire is a fire that is small because it has just started. In the event of a small or incipient fire, the following actions will be taken:

- Evacuate nearby personnel from the area to an upwind location, or to an area not affected by smoke or hazardous decomposition products if an upwind location is not feasible;
- Attempt to extinguish fire using portable fire extinguisher or by smothering;
- Contact EMS for any injuries or exposures to hazardous decomposition products; and
- After the fire has been extinguished, or emergency response personnel have been contacted through **911**, notify the PM, SHM, and the USACE POC.

11.2.20.2 Large Fire / Explosion

An explosion, large fire, or a small fire that cannot be extinguished is beyond the first line capabilities personnel. Professional emergency response personnel would be needed to provide emergency assistance for these types of incidents. In the event of a large fire,

explosion, or a small fire that cannot be extinguished, the following minimum actions will be taken:

- Evacuate all personnel from the site to an upwind location, or to an area not affected by smoke or hazardous decomposition products if an upwind location is not feasible;
- Perform a roll call to account for all site personnel;
- Contact **911** for fire response, and for any injuries or exposures to hazardous decomposition products; and
- After emergency response personnel have been contacted through **911**, notify the PM, SHM, and the USACE POC.

11.2.21 Posting of Emergency Telephone Numbers

Emergency telephone numbers are provided in **Attachment A**. A copy will be kept in each work vehicle.

11.2.22 Man Overboard/Abandon Ship

Not applicable.

11.2.23 Medical Support

Refer to **Section 9.0**, Medical Support.

11.3 Prevention of Alcohol and Drug Abuse Plan

The Drug and Alcohol policy is provided as **Appendix 6**. All project personnel are required to read and abide the policy. The policy will be posted at the job site in the office. The team complies with all applicable federal drug testing program requirements including the training of staff for reasonable suspicion recognition.

11.4 Site Sanitation Plan

The following sanitation provisions will be established and maintained for the duration of this project:

- Drinking water: An adequate supply of cool water will be supplied and will be kept in water coolers in the SZ on-site. Individual bottles of drinking water may be used instead of water coolers. The water cooler will be kept closed. Personnel will be instructed to wash their face and hands prior to drinking;
- Non-Potable Water: Non-potable water maintained at the project site and all outlets dispensing non-potable water shall be labeled as such;
- Personal Hygiene: Sanitary hand wipes or soap and water for washing will be provided;
- Toilet Facilities: A chemical toilet will be available on-site. The toilet will be equipped with toilet paper, toilet paper holder, locking door, and adequate ventilation; and
- Waste Disposal: A trash receptacle will be present in the SZ for the disposal of hand drying materials, any disposable PPE, and other generated site debris.

11.5 Access and Haul Road Plan

Not applicable.

11.6 Respiratory Protection Plan

Refer to **Section 10.7**, Respiratory Protection.

11.7 Health Hazard Control Program

Based on available site historical information, no radiological hazards are expected. The potential hazards associated with the project include chemical, physical, and biological hazards. The potential for encountering chemical hazards depends on the types and quantities of chemicals present and the type of work being performed. The potential for encountering physical and biological hazards depends on the type of work being performed. The following sections will describe the site-specific chemical, physical, and biological hazards, and their control measures.

11.7.1 Chemical Hazards

The main routes of exposure for site personnel include:

- Inhalation of contaminant vapors;
- Inhalation of contaminated particulate matter;
- Ingestion of contaminated material; or
- Dermal absorption of contaminated material.

Due to the nature of the contaminants, local features, and type of site activities planned:

- There is a low to moderate potential for inhalation of contaminant vapors or contaminated particulate matter, including wind-blown particulate matter;
- There is a low potential for ingestion of contaminated material; and
- There is a moderate potential for dermal contact with contaminated material.

Site personnel can reduce their exposure potential by:

- Using the proper PPE; and
- Practicing contamination avoidance, following proper decontamination procedures, and observing good personal hygiene.

11.7.2 Contaminants of Concern

Refer to **Attachment A**, Site Safety and Health Plan.

11.7.3 Chemical Data

In order to protect site personnel from the hazards associated with potential contaminants, a personnel protection and monitoring program will be implemented to control potential exposures. The following sections provide general chemical hazard information for potential site contaminants.

11.7.3.1 Volatile Organic Compounds

Many of the volatile organic compounds (VOCs) are chlorinated solvents that contain at least one chlorine atom. In contrast to non-halogenated organic solvents, chlorinated solvents are denser than water and are not flammable. Based on toxicology, metabolism, animal studies, and human studies, occupational exposure to chlorinated aliphatic solvents (methanes, ethanes, and ethenes) has been associated with numerous adverse health effects, including central nervous system (CNS), reproductive, liver, and kidney toxicity, and carcinogenicity.

11.7.3.2 Petroleum Hydrocarbons

Petroleum Hydrocarbons are a broad family of several hundred chemical compounds that originally come from crude oil. They are called hydrocarbons because almost all of them are made entirely from hydrogen and carbon. Crude oils can vary in how much of each chemical they contain, and so can the petroleum products that are made from crude oils. Most products that contain petroleum hydrocarbons will burn. Some are clear or light-colored liquids that evaporate easily, and others are thick, dark liquids or semi-solids that do not evaporate. Many of these products have characteristic gasoline, kerosene, or oily odors.

Petroleum Hydrocarbons can enter and leave your body when you breathe it in air; swallow it in water, food, or soil; or touch it. Most components will enter your bloodstream rapidly when you breathe them as a vapor or mist or when you swallow them. Some compounds are widely distributed by the blood throughout your body and quickly break down into less harmful chemicals. Others may break down into more harmful chemicals. Other compounds are slowly distributed by the blood to other parts of the body and do not readily break down. When you touch petroleum hydrocarbons compounds, they are absorbed more slowly and to a lesser extent than when you breathe or swallow them. Most compounds leave your body through urine or when you exhale air containing the compounds. The compounds in different petroleum hydrocarbon fractions affect the body in different ways. Some of the compounds, particularly the smaller compounds such as benzene, toluene, and xylene (which are present in gasoline), can affect the human CNS. If exposures are high enough, death can occur. Swallowing some petroleum products such as gasoline and kerosene causes irritation of the throat and stomach, CNS depression, difficulty breathing, and pneumonia from breathing liquid into the lungs. The compounds in some fractions can also affect the blood, immune system, liver, spleen, kidneys, developing fetus, and lungs. Certain compounds can be irritating to the skin and eyes. Other compounds, such as some mineral oils, are not very toxic and are used in foods.

11.7.3.3 Heavy Metals

Toxic metals are individual metals and metal compounds that negatively affect people's health. In very small amounts many of these metals are necessary to support life. However, in larger amounts they become toxic. They may build up in biological systems and become a significant health hazard. Inhalation of respirable dust particles in soil, groundwater, aerosol mists, or ingestion provides the primary pathway for exposures associated with metals. Symptoms of overexposure include increased incidence of respiratory illnesses, mental disturbances, loss of coordination, and respiratory irritation. Metals have the potential for bioaccumulation in various target organs within the body.

Chronic exposure to elemental lead may cause the following symptoms: anemia, dullness, restlessness, irritability, headaches, muscular tremors, and loss of memory. Acute overexposure to lead may cause permanent CNS damage. Elemental lead poses a potential health risk through dermal exposure to lead contaminated waste or soil. Inhalation of lead-contaminated dust is also a potential route of exposure.

Breathing in high levels of hexavalent chromium can cause irritation to the nose and throat. Symptoms may include runny nose, sneezing, coughing, itching and a burning sensation. Repeated or prolonged exposure can cause sores to develop in the nose and result in nosebleeds. If the damage is severe, the nasal septum (wall separating the nasal passages) develops a hole in it (perforation).

Allergies to hexavalent chromium may develop; inhaling chromate compounds may cause asthma symptoms (e.g. wheezing, shortness of breath), and/or an allergic skin reaction. Once a person becomes allergic to chromium, brief skin contact causes swelling and a red, itchy rash

that becomes crusty and thickened with prolonged exposure. The skin allergy is long-lasting and more severe with repeated skin contact. Direct skin contact with hexavalent chromium may cause a non-allergic skin irritation, and contact with non-intact skin may also lead to chrome ulcers, which heal slowly and leave scars.

11.7.3.4 Semi-Volatile Organic Compounds

Semi-volatile organic compounds (SVOCs) are of concern because some compounds have been identified as carcinogenic, mutagenic, and teratogenic. SVOCs are lipophilic, meaning they mix more easily with oil than water. The larger compounds are less water-soluble and less volatile. Because of these properties, SVOCs in the environment are found primarily in soil, sediment and oily substances, as opposed to in water or air. Though the inhalation potential of these compounds is less than VOCs, the possibility of an inhalation exposure still exists as they can be in particulate matter suspended in air. The possibility of skin absorption also exists. SVOCs may cause irritation to the eyes, upper respiratory system, or skin after prolonged or repeated exposure. Overexposure to high levels of may cause dizziness, headache, nausea, drowsiness, and other nervous system effects.

11.7.3.5 Polychlorinated Biphenyls

Polychlorinated biphenyls (PCBs) are a group of manufactured organic chemicals that contain 209 individual chlorinated chemicals (known as congeners). Concentrated PCBs are either oily liquids or solids and are colorless to light yellow in color. They have no known smell or taste. There are no known natural sources of PCBs. Some commercial PCB mixtures are known in the United States by their industrial trade name, Aroclor. PCBs have been demonstrated to cause a variety of adverse health effects. PCBs have been shown to cause cancer in animals. PCBs have also been shown to cause a number of serious non-cancer health effects in animals, including effects on the immune system, reproductive system, nervous system, endocrine system and other health effects. Studies in humans provide supportive evidence for potential carcinogenic and non-carcinogenic effects of PCBs.

11.7.3.6 Pesticides

Pesticides do not occur naturally in the environment. They are typically extremely persistent pollutants as they do not easily break down. Therefore they tend to biomagnify as they are passed along the food chain. Long-term exposure has proven toxic to a very wide range of animals including humans, far greater than to the original insect targets. Pesticides have been linked to health problems such as Parkinson's, breast cancer, and immune, reproductive, and nervous system damage. Moderate levels of exposure can cause headaches, dizziness, irritability, vomiting, and uncontrolled muscle movements.

11.7.3.7 Asbestos

Asbestos is a group of six naturally-occurring minerals that can break apart into fibers. Friable asbestos is a term used to describe any asbestos-containing material that when dry, can be easily crumbled or pulverized to powder by hand. Some common examples of friable asbestos are acoustic ceilings and tiles, many types of plasters, wallboard, joint compound or “mud” and thermal insulation for water heaters and pipes.

When asbestos is crushed, it disperses a dusting of microscopic fibers in the air that can remain for very long periods of time. These fibers can be unknowingly inhaled and permanently lodged in lung and other body tissues, yet symptoms might not appear for 20 years or more. Inhaling the fibers has been linked to cancer and asbestosis, a chronic lung disease similar in symptoms to emphysema.

11.7.3.8 Carbon Monoxide

Carbon monoxide (CO) is a colorless, odorless, and tasteless gas which is slightly lighter than air. It is highly toxic to humans and animals in higher quantities. It combines with hemoglobin to produce carboxyhemoglobin, which is ineffective for delivering oxygen to bodily tissues. The most common symptoms of CO poisoning may resemble other types of poisonings and infections, including symptoms such as headache, nausea, vomiting, dizziness, fatigue and a feeling of weakness. Neurological signs include confusion, disorientation, visual disturbance, syncope and seizures. Exposures to carbon monoxide may cause significant damage to the heart and CNS.

11.7.3.9 Diesel Exhaust

Emissions from diesel-fueled engines come from internal combustion engines burning diesel fuel and are made up of a complex mixture of thousands of gases, vapors, and fine particles. Particulate matter from diesel-fueled engine emissions is small enough to be inhaled deep into the lungs. Several studies indicate that workers exposed to diesel exhaust over a number of years are more likely to contract lung cancer in addition to other maladies. Not only is the particulate matter associated with cancer, but the gases in diesel exhaust are suspect as well.

11.7.3.10 Silica

Silicosis, an irreversible but preventable disease, is the illness most closely associated with occupational exposure to silica dust. Occupational exposures to respirable crystalline silica are associated with the development of silicosis, lung cancer, pulmonary tuberculosis, and airways diseases. These exposures may also be related to the development of autoimmune disorders, chronic renal disease, and other adverse health effects.

11.7.3.11 Nuisance Dusts

Excessive concentrations may seriously reduce visibility, cause unpleasant deposits in eyes, ears, and nasal passages, or cause injury to the skin or mucous membranes by chemical or mechanical action. There is no particulate that does not provoke some response when inhaled in sufficient amounts.

11.7.4 Carbon Dioxide

Carbon Dioxide (CO_2), one of the major components of landfill gas, is asphyxiating and causes adverse health effects at relatively low concentrations. It is relatively nontoxic and noncombustible. In its pure form, it is odorless, colorless, and heavier than air.

11.7.5 Methane

Methane (CH_4) is the other major components of landfill gas. Methane is a colorless odorless gas that is easily ignited and has vapors lighter than air. Methane is not toxic below the lower explosive limit (LEL) of 5 percent (50,000 parts per million [ppm]). However, when methane is present at high concentrations, it acts as a simple asphyxiant. Simple asphyxiants displace oxygen in the air and can cause symptoms of oxygen deprivation (asphyxiation).

11.7.6 Physical Hazards

A variety of physical hazards may be present during site activities. These hazards are similar to any construction-type project, and are generally familiar to most site workers. Task-specific hazards will be covered during site safety briefings.

11.7.6.1 Work at Heights

Work may be performed at a level that is greater than 6 feet above a lower level. Ladders are allowed for this project but must be inspected by the SSHO prior to use. Employees who perform work while on a ladder are not required to wear fall protection as long as the ladder is used properly. Place ladder on level ground. Don't stand higher than the third step below the top of the ladder. Personnel are required to use fall protection equipment any time they are at a height of 6 feet above a lower level if not on a ladder.

The SSHO shall review ladder safety and training during the initial and daily safety and health tailgate meetings. The SSHO is responsible for inspecting, maintaining, and providing training on fall protection equipment (i.e. body harnesses, lanyard, fall-arrestors, etc.).

11.7.6.2 Drum/Container Handling Procedures

Drums and containers used during site activities will meet appropriate Department of Transportation (DOT), OSHA, and Environmental Protection Agency (EPA) regulations for the products or waste they will contain. Employees participating in activities involving drum or container use will be trained in the hazards associated with the drum activities. Drums and containers will be inspected and their integrity will be verified prior to being moved.

Drums or containers that cannot be inspected prior to movement because of storage conditions will be positioned in an accessible location and inspected prior to further handling. Activities on-site will be organized to minimize the amount of drum or container movement. Drums or containers that cannot be moved without failure will be emptied into a second container. Refer to **Section 11.2.10**, Hazardous Substance Spill or Release for drum, and container spill contingency procedures.

11.7.6.3 Excavation/Trenching

An excavation is any man-made cavity or depression. Depending on its depth, width, and the presence of a hazardous atmosphere, an excavation may also be considered to be a confined space. Excavations are defined to include trenching. A trench is a narrow excavation in which the depth is greater than the width, and width is not greater than 15 feet. Trenches excavated deeper than 4 feet are considered confined spaces. The site activities currently scheduled for the project site include excavation activities.

All excavations will be performed in accordance with 29 CFR 1926 Subpart P. Prior to starting any excavation, the possibility of the presence of underground pipelines, electric wires, conduits, or vessels containing material under pressure will be investigated. The SSHO will coordinate with the USACE POC and local utility locators to locate and shut-off existing utilities. All surface encumbrances that will create a hazard to workers will be removed or supported.

An OSHA-competent person will inspect the excavations, adjacent areas, and protective systems on the following schedule:

- Daily and prior to work in or around the excavation;
- After every rain storm or other hazard-increasing occurrence; and
- As needed throughout the work as conditions change.

If the inspector notes a hazardous condition, all endangered workers must be immediately removed from the hazard, and all work at the excavation stopped until the necessary corrections have been made. A Daily Excavation/Trench Inspection Report form is located in **Appendix 3**, "Inspection Forms."

The following safety controls will be implemented during excavation activities:

- If the excavation endangers the stability of adjacent structures, then support systems such as shoring, bracing, or underpinning will be provided;
- Excavated soil will be placed a minimum 2 feet from the edge of the excavation. Stockpiled materials should not be placed closer than half the maximum depth of the excavation;
- Due to the limited work area, all site personnel entering and working within the site will be required to wear high-visibility “traffic” vests when heavy equipment/motor vehicles are in use;
- When mobile equipment is operated near an excavation, or required to approach the edge of an excavation, a warning system (e.g. barricades, hand signals, mechanical signals, stop logs) will be used;
- Workers are not permitted underneath loads handled by lifting or excavating equipment; and
- Prior to exiting the site, the SSHO will ensure that temporary orange fencing, caution tape, or similar barrier is set up around any open excavations to prevent unauthorized personnel from falling into an excavation during non-working hours. The barriers will remain in place until the excavation has been backfilled any time the excavation is unsupervised.

11.7.6.4 Flammable and Combustible Materials

Handling, storage, and use of flammable and combustible liquids will be in compliance with OSHA 29 CFR 1926.152. All flammable and combustible liquids will be stored in a well-ventilated area, and away from excessive heat and direct sunlight. These liquids will not be stored in areas used for exits, stairways, or aisles. Material that reacts with water will not be stored near flammable or combustible liquids. All sources of ignition are prohibited in this area, including smoking, cutting and welding, hot surfaces, open flames, sparks (static, electrical, and mechanical), and frictional heat.

"Flammable Liquids" and "No Smoking or Open Flames" signs will be posted in the storage area. At least one portable fire extinguisher rated not less than 20-B will be located within 10 feet from the entrance to the storage area, and at least one similar fire extinguisher will be located between 25 and 75 feet outside the storage area.

Flammable and combustible liquids should only be handled in areas that have adequate ventilation. Workers are not permitted to use liquids having a flashpoint less than 100 degrees Fahrenheit (°F) as a cleaning/degreasing fluid. Workers will change as soon as possible if flammable or combustible liquid is spilled on clothing.

Dispensing areas for transfer of more than 5 gallons of flammable liquids will be separated from hot work areas by at least 25 feet. Spills in these areas will be controlled using drainage, diking, or absorbent material. Flammable liquids will only be transferred when the two containers are electrically interconnected (i.e. bonded). When dispensing flammable and combustible liquids into smaller portable containers, only approved safety containers equipped with back flash arresters will be used.

11.7.6.5 Hand/Power Tools

All hand and power tools will be maintained in a safe condition and in good repair. Hand and power tools will be used in accordance with 29 CFR 1926 Subpart I - 1926.300 through 1926.307. Neither employees nor subcontractors will issue unsafe tools, nor are workers permitted to bring unsafe tools on-site.

All tools will be used, inspected, and maintained in accordance with the manufacturer's instructions. Throwing tools or dropping tools to lower levels is prohibited. Hand and power tools will be inspected, tested, and determined to be in safe operating condition prior to each use. Any tool that fails an inspection will be immediately removed from service and either discarded or tagged with a "Do Not Use" sign until repairs are made.

Workers using hand and power tools who are exposed to falling, flying, abrasive, or splashing hazards are required to wear PPE; eye protection must always be worn when working on-site. Additional eye and face protection (i.e. safety goggles or face shields) may also be required when working with specific hand and power tools. Workers using tools in areas where there is a head injury hazard will wear approved head protection.

Hearing protection will always be worn when working with power tools. Workers using tools that may subject their hands to an injury, such as cuts, abrasions, punctures, or burns, will wear protective gloves. Loose or frayed clothing, dangling jewelry, or loose long hair will not be worn when working with power tools.

Electric power-operated tools will be double insulated or grounded, and equipped with an on/off switch. Guards must be provided to protect the operator and other nearby workers from hazards such as in-going nip points, rotating parts, flying chips, and sparks. All reciprocating, rotating, and moving parts of tools will be guarded if contact is possible. Removing machine guards is prohibited.

11.7.6.6 Heat Stress/Cold Stress

The project field activities are currently year-round so heat stress is a concern. If unusual conditions occur or field work runs longer than expected cold stress could be a potential concern. The SSHO is responsible for monitoring heat and cold stress.

Heat stress – is a significant potential hazard during the warmer months. Heat stress may manifest itself as: heat rash (prickly heat), transient heat fatigue, heat cramps, fainting (heat syncope), heat exhaustion, or heat stroke. **Section 11.14.1** outlines the first aid procedures for heat stress illnesses.

Cold stress – is a danger at low temperatures and when the wind chill factor is low. Cold stress is generally described as a local cooling (frost nip, frostbite, and freezing) or a general cooling (hypothermia). **Section 11.14.2** outlines the first aid procedures for cold stress injuries.

Personnel working outdoors in temperatures at or below freezing may be subject to local cooling. Areas of the body that have a high surface area-to-volume ratio, such as fingers, toes, and ears, are the most susceptible.

11.7.6.7 Heavy Equipment and Motor Vehicle Operation

Only qualified personnel will operate heavy equipment and motor vehicles. All heavy equipment and motor vehicles will be operated in accordance with the manufacturer's instructions; OSHA 29 CFR 1926 Subpart O. Operators will follow these rules:

- Seat belts will be worn when operating moving equipment;
- The on-site speed limit is no more than 25 miles per hour (mph) unless otherwise posted;
- Operators will not leave their equipment unattended while it is running;
- Whenever equipment is parked, the parking brake will be set. If the equipment is parked on an incline, in addition to setting the parking brake, the wheels will also be chocked;
- Operators will be trained and experienced in the use of their equipment;

- Vehicles or equipment will not be operated in a careless or unsafe manner;
- Passengers or "riders" are not allowed on equipment; and
- The operator shall use means of ingress and egress as designed to get on and off the equipment. Do not jump directly off the equipment to the ground.

Required equipment features include:

- All equipment and vehicles will have an audible backup alarm and an audible warning device (i.e. a horn);
- Each vehicle and piece of equipment will have a portable fire extinguisher rated not less than 2-A:10-B:C; and
- Equipment will be properly guarded.

When working with moving equipment:

- All personnel will stay clear of the operational area of the equipment. Workers are not permitted to stand directly behind or underneath any piece of equipment or load (i.e. crane load, excavator bucket);
- Work areas will be adequately illuminated;
- Workers are prohibited from riding in equipment buckets and booms; and
- All site personnel entering and working within the work area will be required to wear high-visibility “traffic” vests when heavy equipment/motor vehicles are in use.

11.7.6.8 Illumination

Guidelines in Table D-3, OSHA 1926.56, “Illumination,” will be followed in determining minimum lighting requirements. Portable floodlights or other similar temporary lights will be used, as needed, to illuminate the work area. All artificial lights, motors, extension cords, connectors, switches, etc., will meet the requirements of National Fire Protection Association (NFPA) 70. Fieldwork will be completed during daylight hours (i.e. between sunrise and sunset).

11.7.6.9 Manual Material Handling

Movement of materials and equipment may lead to muscle strains, pinching injuries to hands and fingers, and crushing-type injuries. Risks of these injuries can be controlled by wearing appropriate footwear, seeking assistance when lifting or moving heavy objects, using mechanical aids to move or lift heavy objects, avoiding overexertion and use of excessive force, and taking rest breaks as needed.

Back injuries are among the leading occupational injuries reported by industrial workers. Using proper manual lifting techniques can reduce back injuries such as pulls and disc impairments. Leg muscles are stronger than back muscles, so workers should lift with their legs and not with their back.

Guidelines for safe lifting, shoveling, and digging include:

- If the load is too heavy, then do not lift it alone. Lifting is always easier when performed with another person. Assistance should always be used when it is available;
- Use a pushcart or other material-handling device whenever possible;
- Pushing a load is easier on the back than pulling a load;
- Do not lift objects over your head; and
- Pace yourself to avoid fatigue when doing heavy work for a long period of time. Rotate the task among workers to share the heavy work.

11.7.6.10 Noise

Noise is a potential hazard associated with the operation of heavy equipment, power tools, pumps, or generators. The SSHO will evaluate high noise operations. As a general practice, hearing protection will be worn when operating heavy equipment, power tools, generators, or when verbal communication is difficult at an arm's length away.

11.7.6.11 Slipping/Tripping Hazards

As with any ERS-type project, uneven work surfaces and other slipping or tripping hazards may be present. These hazards are exacerbated when walking/working surfaces become muddy or wet. As much as possible, site workers will keep walking/working surfaces free from excessive water and mud, and avoid climbing on uneven terrain. High traction footwear should be used if the work area becomes muddy. Proper site housekeeping, removal of trash, and orderly stacking and removal of materials will also reduce slipping and tripping hazards.

11.7.6.12 Solar Radiation

Hazards from excessive exposure to the sun include sunburn, cancers, and eye damage from the ultraviolet (UV) radiation. Recommended protection for UV Radiation is a wide-brimmed hat, long pants, sleeved shirts, and safety glasses with UV protection. Sunscreens with a high Sun Protection Factor (SPF) should be applied to exposed skin.

11.7.6.13 Utility Location

11.7.6.13.1 Overhead utilities

All overhead utility lines are treated as energized until determined as de-energized by testing. The PM will notify the utility company when working near or moving equipment in close proximity under utility lines. Work adjacent to overhead utility lines will not be permitted until one of the following three conditions is met: the power has been shut off, insulating barriers have been erected, or the minimum required safe clearance has been marked.

If overhead utility lines cannot be shut off, then insulating barriers (which are not part of/attached to the equipment) will be erected to prevent physical contact with the line, and/or a minimum safe clearance work zone will be set-up. All parts of heavy equipment and motor vehicles must have the capability of staying outside the minimum clearance of the energized overhead lines as specified in EM 385-1-1 Section 11.F "Operations Adjacent to Overhead Lines."

Nearby overhead power lines shall be located and evaluated to determine the voltage of the line. Nearby lines shall be de-energized, guarded, or minimum clearance distances shall be maintained at all times as shown in **Table 11-9**.

Table 11-9 Overhead Line Clearance Distances

Nominal Voltage (kV)	Clearance Distance (feet)
Up to 50	10
51 – 200	15
201 – 350	20
351 – 500	25
501 – 750	35
751 – 1,000	45
> 1,000	As determined by Professional Engineer (PE).

11.7.6.13.2 Underground utilities

The major hazards involved with hitting an underground utility line include electric shock/electrocution (electric utility line), fire/explosion (gas utility line), trench cave-in (water/sewer utility line), heat burns (high pressure steam utility line), and costs associated with repair and disruption of the utility's service.

One of the most important elements of site preparation is to locate all underground utility services. The possibility of the presence of underground utilities, pipelines, or vessels containing material under pressure will be investigated prior to the start of any invasive work. Underground utility clearance and dig permits will be obtained in conjunction with the USACE POC. The SSHO will document the utility clearance. A list of utility contacts is provided in **Table 11-10**.

Table 11-10 Utility Contacts

Company	Description	Number
Miss Utility	Call before you dig; utility clearance.	800-257-7777; or 811
JBA Dig Permit	Compile dig permit and obtain appropriate signatures.	See Site-Specific Dig Permit, if applicable
Pepco	Contact, scrape, dent, nick or otherwise damage any electric line.	877-737-2662; **911**
Washington Gas	Contact, scrape, dent, nick or otherwise damage any gas line.	800-752-7520; or 703-750-1400; **911**

11.7.6.14 Weather-Related Hazards

Weather conditions are a part of the daily safety briefing. During Severe weather, project personnel will seek shelter in an appropriate location (i.e. building or vehicle). Weather-related hazards include the potential for heat or cold stress, electrical storms, ice/snow storms, hurricanes, tornadoes, high winds and dust storms, limited visibility, and treacherous weather-related working conditions.

These hazards correlate with the season in which site activities occur. Outside work will be suspended during electrical storms. In the event of other adverse weather conditions, the SSHO will determine if work can continue on-site without endangering the safety and health of site personnel.

11.7.6.14.1 Thunderstorm and Lightning Activity

The safest shelter location during a thunderstorm and lightning activity is a large, enclosed building. If lightning strikes this type of building it will typically travel through the wiring or plumbing into the ground. Once inside a safe building, stay away from the plumbing and electronic equipment. The second safest shelter location is in an enclosed vehicle with all the doors and windows closed. Do not touch any metal surfaces, and do not use any electronic devices, including cell phones.

Site personnel will seek shelter when dark threatening clouds are seen developing overhead, lightning is seen, or thunder is heard. Personnel must remain protected for 30 minutes after the last visible lightning strike or audible thunder clap. No place is absolutely safe from severe weather; however, some places are safer than others:

- Large enclosed structures tend to be much safer than smaller or open structures;
- The risk for lightning injury depends on whether the structure incorporates lightning protection, construction materials used, and the size of the structure;
- In general, fully enclosed metal vehicles such as cars, trucks, buses, vans, etc. with the windows rolled up provide good shelter from many weather conditions;
- Avoid being in or near high places and open fields, isolated trees, rain or picnic shelters, communications towers, flagpoles, light poles, bleachers (metal or wood), metal fences, water (lakes, streams, rivers, etc.); and
- When inside a building, avoid the use of the telephone, washing your hands, or any contact with conductive surfaces with exposure to the outside such as metal door or window frames, electrical wiring, telephone wiring, cable TV wiring, plumbing, etc. if lightning is a factor.

11.7.6.14.2 Tornadoes

Tornadoes always come out of severe thunderstorms. They may be stationary or travel at speeds up to 70 mph, and generally travel from southwest to northeast. When severe thunderstorms are in the area, site personnel will watch the sky, and the SSHO will monitor the weather advisories.

A Tornado Watch means the weather conditions are favorable for the development of a tornado. A Tornado Warning means a tornado has been sighted or indicated on radar. If you are in the warned area, seek shelter immediately. The general rule for tornadoes is “go low and get low:”

Go to the lowest level of a sturdy building structure. The best shelter from a tornado is a basement. If a basement is not available, go to an inside room, without windows.

- Take shelter in the lowest level;
- Stay away from windows;
- Crouch in a low position;
- Protect your body from flying debris with a blanket or jacket; and
- Protect your head.

If you are in a trailer, vehicle, or outside, try to get to a sturdy building for shelter. Trailers and vehicles are extremely unsafe during tornadoes; seek shelter elsewhere. If you cannot get to a sturdy building, then lie flat in a low area (e.g. ditch, depression) and use your hands to cover the back of your head and neck. Environmental “clues” that may indicate an approaching tornado include:

- A dark and often greenish sky;
- Large hail; and/or
- A loud roar similar to a freight train.

11.7.6.14.3 Hurricanes

The Atlantic hurricane season runs from June 1 through November 30, with peak activity occurring August through October. While hurricanes can produce extremely powerful winds and torrential rain, they are also able to produce high waves and damaging storm surge as well as spawning tornadoes. They develop over large bodies of warm water, and lose their strength if they move over land due to increased surface friction and loss of the warm ocean as an energy source.

A Hurricane / Tropical Storm watch means that storm conditions are possible in the watch area within 48 hours. Take the following precautions in preparation for the storm:

- Listen frequently to National Oceanic and Atmospheric Administration (NOAA) Weather Radio, TV, or radio for bulletins of a storms progress and potential path;
- Fuel and ensure your vehicle(s) are in good operating condition;
- Secure equipment especially light weight objects that could become projectiles in strong winds; and
- Contact PM and/or SSHO to reschedule work and initiate evacuation details.

A Hurricane / Tropical Storm Warning means that storm conditions are expected in the warning area within 36 hours. If a warning is issued, prepare to evacuate:

- If an evacuation order is given, be prepared to leave immediately;
- Make arrangements with the PM for your final destination; and
- Consider the travel path of the hurricane in choosing your destination and travel route.

11.7.6.15 Explosive Ordnance and Explosives

Hazards from MEC include fragmentation, thermal burns, and concussions that would result from an unintentional detonation. Based on a review of the historical information, MEC is not anticipated at the work sites. However, if suspected MEC is encountered during ERS activities, all work will immediately cease.

11.7.7 Biological Hazards

11.7.7.1 General Wildlife

Wildlife may attack if disturbed or threatened. If wildlife is sighted during field work, especially if an animal appears to be disoriented, aggressive, or exhibits other strange behavior, proceed from the area to the site vehicles to avoid the animal. The possibility of encountering wildlife is moderate and will be communicated to all site workers during the initial site-specific safety training. Workers will be warned to avoid wildlife and to report any encounters.

Biological hazards, which may be found on-site, include insects, arachnids (e.g. spiders, scorpions), and plants. Several varieties of snakes and other wildlife are also common hazards in this area. Employee awareness and the safe work practices outlined in the following paragraphs should reduce the risk associated with these hazards to acceptable levels.

11.7.7.2 Biting and Stinging Insects

Biting and stinging insects (e.g. bees, wasps, ticks, mites and spiders) may be encountered on-site. The Field Operations Lead or SSHO (as applicable) will encourage the use of insect repellents, if deemed necessary. The biting insects of greatest concern are spiders, especially the black widow and the brown recluse spider. These spiders are of special concern due to the significant adverse health effects that can be caused by their bite.

11.7.7.3 Bees, Hornets and Wasps

Contact with stinging insects (e.g. bees, hornets, velvet ants, and wasps) may result in site personnel experiencing adverse health effects that range from mild discomfort to life-threatening. Therefore, stinging insects present a serious hazard to site personnel, and extreme caution must be exercised whenever site and weather conditions increase the risk of encountering stinging insects. Some of the factors that are related to stinging insects that increase the degree of risk associated with accidental contact are as follows:

- The nests for these insects are frequently found in remote wooded, grassy areas where many waste sites are located;
- The nests can be situated in trees, rocks, and bushes or in the ground, and are usually difficult to see;
- Accidental contact with these insects is highly probable, especially during warm weather conditions when the insects are most active;
- If a site worker accidentally disturbs a nest, the worker may be inflicted with multiple stings, causing extreme pain and swelling which can leave the worker incapacitated and in need of medical attention; and
- Some people are hypersensitive to the toxins injected by a sting, and when stung, experience a violent and immediate allergic reaction resulting in a life-threatening condition known as anaphylactic shock. Anaphylactic shock manifests itself very rapidly and is characterized by extreme swelling of the body, eyes, face, mouth and respiratory passages.

The hypersensitivity needed to cause anaphylactic shock can, in some people, accumulate over time and exposure; therefore, even if someone has been stung previously, and has not experienced an allergic reaction, there is no guarantee that they will not have an allergic reaction upon receipt of another sting.

11.7.7.4 Ticks

The Center for Disease Control (CDC) has noted the increase of Lyme disease and Rocky Mountain spotted fever (RMSF) caused by bites from infected ticks that live in and near wooded areas, grass, and brush. Ticks are small, ranging from the size of a comma up to about one quarter inch (**Figure 11-1**). They are sometimes difficult to see. When embedded in the skin, they may look like a freckle. The tick season extends from spring through summer.

Lyme disease has occurred in 49 states, with the heaviest concentrations in the Northeast (Connecticut, Massachusetts, New Jersey, New York, and Pennsylvania), the upper Midwest (Minnesota and Wisconsin), and along the northern California coast. It is caused by deer ticks and the lone star ticks that become infected with spirochetes. Female deer ticks are about one quarter inch in size, and are black and brick red in color. Male deer ticks are smaller, and completely black. Lone star ticks are larger and chestnut brown in color.

RMSF has occurred in at least 36 states, with the heaviest concentrations in Oklahoma, North Carolina, South Carolina, and Virginia. It is caused by Rocky Mountain wood ticks and dog ticks that have become infected with rickettsia. Both are black in color.

The first symptoms of either disease are flu-like chills, fever, headache, dizziness, fatigue, stiff neck, and bone pain. If immediately treated by a physician, most individuals recover fully in a short period of time. If not treated, more serious symptoms can occur. If you believe you have been bitten by a tick, or if any of the signs and symptoms noted above appears, contact the SSHO, who will authorize you to visit a physician for an examination and possible treatment.

11.7.7.5 Mites (Chiggers)

Chiggers are small mites that are usually a yellowish to bright red color (**Figure 11-2**). Chiggers may live year-round but are especially active during spring and summer. The larval chigger is the active stage that bites animals and humans, attaching themselves tightly. After secreting digestive enzymes that break down the skin cells, the mite feeds on the liquefied cells. The rash and intense itching associated with chiggers is an allergic reaction to the mite's salivary secretions. Preventive measures used against mosquitoes are effective against chiggers.

Treatments to ease itching include ointments such as calamine lotion, hydrocortisone, and benzocaine.

11.7.7.6 Black Widow Spider

The black widow spider is not aggressive unless agitated when guarding her egg sac. They live in a variety of natural and domestic habitats such as under rocks, wooden boards and in dense plant growth. The female spider is glossy black and marked with a characteristic red hourglass on the underside of the abdomen. The female has a body length of about ½" with a total length of about 1 ½", (**Figure 11-3**).

The male, which is rarely seen, is smaller and has four pairs of red marks along the sides of the abdomen. Young black widow spiders are tan-to-gray in color and have orange and white "racing stripes" on their abdomens.

Black widow spider venom affects the nervous system. The venom causes pain in the lymph nodes. Other symptoms of a severe bite include nausea, elevated blood pressure, sweating, tremors and increased white blood cell counts. The wound may appear as a bluish red spot, surrounded by a whitish area. Victims of a black widow bite may exhibit the following signs or symptoms:

- Sensation of pinprick or minor burning at the time of the bite; and
- Appearance of small punctures (but sometimes none are visible).

After 15 to 60 minutes, intense pain is felt at the site of the bite which spreads quickly, and is followed by profuse sweating, rigid abdominal muscles, muscle spasms, breathing difficulty, slurred speech, poor coordination, dilated pupils and generalized swelling of face and extremities.

11.7.7.7 Snakes

Depending on the time of year of the field work, snakes could be encountered. Personnel should be aware of their surroundings and take particular care when traversing areas that may be inhabited by snakes, such as near rocks, logs, crevices and in holes or pipes (**Figure 11-4** and **Figure 11-5**).

11.7.7.8 Plants Causing Skin Reactions

A number of hazardous plants may be encountered. The ailments associated with these plants range from mild hay fever to contact dermatitis to carcinogenic affects. The plants that present the greatest degree of risk to site personnel (i.e. potential for contact vs. affect produced) are those that produce skin reactions and skin and tissue injury.

The hazardous plants of greatest concern are a variety of poison ivy (**Figure 11-6**), poison oak (**Figure 11-7**), or poison sumac (**Figure 11-8**) found in the project area. Contact with the leaves, stems, or roots of these plants may produce redness, blisters, swelling, and intense burning and itching due to transfer of oils or sap. The victim also may develop an infection should the surface of the skin be broken. The most distinctive features of hazardous plants are readily visible. These plants may grow in all areas around the project site.

Improper treatment of an injury can cause secondary infections to occur. Preventive measures that can prove effective for most site personnel:

- Avoid contact with any hazardous plants on-site;
- Remove gloves prior to touching face, neck, or other exposed areas of the body;

- Wash hands, face or other exposed areas at the beginning of each break period and at the end of each workday; and
- Keeping the skin covered as much as possible (i.e. long pants and long sleeved shirts) in areas where these plants are known to exist will limit some of the potential exposure.

11.7.7.9 Plants Causing Skin and Tissue Injury

Contact with sharp leaves, nettles, and thorns are of special concern to site personnel. This concern stems from the fact that punctures, cuts and even minor scrapes caused by accidental contact may result in non-infectious skin lesions, and the introduction of fungi or bacteria through the skin or eye. This is especially important in light of the fact that the warm moist environment created inside protective clothing is ideal for the propagation of fungal and bacterial infection.

Personnel receiving any of the injuries listed above, even minor scrapes will report it immediately to the SSHO or Field Operations Lead for initial and continued observation and care of the injury. Keeping the skin covered as much as possible (i.e. long pants and long sleeved shirts) in areas where these plants are known to exist will limit much of the potential exposure. If the rash is scratched, secondary infections can occur. The rash usually disappears in one to two weeks in cases of mild exposure and up to three weeks when exposure is severe.

Preventive Measures: The hazardous plants of greatest concern are those varieties found in the project area having the ability to cause redness, blisters, swelling, and intense burning and itching due to punctures, scraps, or lacerations. Improper treatment of an injury can cause secondary infections to occur. Preventive measures that can prove effective for most site personnel are:

- Avoid contact with any hazardous plants on-site;
- Remove gloves prior to touching face, neck, or other exposed areas of the body;
- Wash hands, face or other exposed areas at the beginning of each break period and at the end of each workday; and
- Keeping the skin covered as much as possible (i.e. long pants and long-sleeved shirts) in areas where these plants are known to exist will limit some of the potential exposure.

Figure 11-1 Deer Tick

Figure 11-2 Chigger

Figure 11-3 Black Widow Spider

Figure 11-4 Rattlesnake

Figure 11-5 Copperhead

Figure 11-6 Poison Ivy

Figure 11-7 Poison Oak

Figure 11-8 Poison Sumac

11.8 Hazard Communication Program

The majority of hazardous materials used will be in limited quantities in end-consumer containers such as batteries, spray paint, printer ink, window cleaner and sanitary hand wipes. No MSDSs are required for these items in accordance with 29 CFR 1910.1200(b) (6) (ix).

MSDSs for hazardous materials are included in **Attachment A**.

Chemical containers (primary and secondary) shall be correctly and clearly labeled with the name of the chemical and the hazard(s) associated with that chemical (e.g. flammable, corrosive, etc.).

11.9 Process Safety Management Plan

Not applicable.

11.10 Lead Abatement Plan

Not applicable.

11.11 Asbestos Abatement Plan

Not applicable.

11.12 Radiation Safety Program

11.12.1 X-Ray Radiation

Analytical x-ray equipment makes use of very narrow collimated x-ray beams of high intensity. Exposure of the eyes or the skin of the body to the primary x-ray beam may result in severe radiation burns in a matter of seconds. Localized radiation burns produced by the high intensity primary x-ray beam are the principal hazards associated with the use of analytical x-ray equipment.

A hazard may also exist from exposure to scattered radiation. Scattered radiation is produced when the primary beam strikes collimators, samples, beam stops or shielding. The intensity of the scattered radiation is a couple of orders of magnitude less than that of the primary beam. It is possible for these scattered radiation fields to result in exposure. The X-Ray Fluorescence (XRF) operator is required to:

- Successfully complete a radiation safety course and be on the authorized user list;
- Review the X-Ray Radiation Protection Program (RPP) prior to operation (**Attachment A**);
- Review radiation safety during tailgate safety meetings (e.g. time, distance, shielding);
- Ensure a regulatory permit has been issued and is valid;
- Ensure notice has been given to regulatory agent on expected use;
- Ensure required postings are posted; and
- Perform personnel monitoring as required by the installation or regulatory agency.

11.13 Abrasive Blasting

Not applicable.

11.14 Heat and Cold Stress Monitoring Plan

The SSHO is responsible to ensure temperature stress controls are adequate for the site conditions and tasks. All employees, and specifically the SSHO, are empowered and expected to stop or modify work and take any precautionary measure to prevent temperature-related illnesses.

The ambient air temperature will be measured during the day. If the temperatures reach or exceed 70°F or reach or fall below 40°F, temperature stress controls will be implemented. All temperature monitoring results, physiological monitoring results, and temperature stress controls will be documented in field records using logbooks.

Monitoring is no substitute for common sense. If workers are affected by temperature extremes take appropriate measure such as adjusting work schedules, increasing fluid intake or drinking warm liquids.

11.14.1 Heat Stress

Heat stress is one of the most common (and potentially serious) illnesses that affect site workers. When site personnel are engaged in operations involving hot environments, a number of physiological responses can occur that may seriously affect the health and safety of the workers. These effects can be eliminated or controlled through the use of a comprehensive heat stress prevention and monitoring program.

Individuals vary in their susceptibility and degree of response to stress induced by increased body heat. Heat stress can result in health effects ranging from transient heat fatigue to serious illness or death. Heat stress is caused by a number of interacting factors including environmental conditions, clothing, workload, and the individual characteristics of the worker. Because heat stress is probably one of the most common (and potentially serious) illnesses at work sites, regular physiological or area monitoring (as appropriate) and other preventive precautions are vital.

Factors that may predispose a worker to heat stress include:

- Lack of physical fitness;
- Lack of acclimatization to hot environments;
- Degree of hydration;
- Level of obesity;
- Current health (i.e. having an infection, chronic disease, diarrhea, etc.);
- Alcohol or drug use;
- The worker’s age and sex; and
- Prior history of heat stress.

The amount and type of PPE worn directly influences reduced work tolerance and increases the risk of excessive heat stress. PPE adds weight and bulk, severely reduces the body’s access to normal heat exchange mechanisms (evaporation, convection, and radiation), and increases energy expenditure. When selecting PPE, each item’s benefit must carefully be evaluated in relation to its potential for increasing the risk of heat stress.

Once PPE is selected, the safe duration of work/rest periods are determined based on the:

- Anticipated work rate;
- Ambient temperature and other environmental factors;
- Type of protective ensemble; and
- Individual worker characteristics and fitness.

Sweating does not cool the body unless moisture is removed from the body. The use of PPE reduces the body's ability to eliminate large quantities of heat because the evaporation of sweat is decreased. The body's effort to maintain an acceptable temperature may become impaired

and may cause heat stress. Increased body temperature and physical discomfort also promote irritability and a decreased attention to the performance of hazardous tasks.

For this project, Level D PPE will typically be utilized, thus providing minimal increase in the potential for heat stress. Level D PPE is defined as standard work clothes with sturdy work boots, long pants, short or long sleeve shirt as applicable, safety glasses, appropriate gloves, hard hats and safety boots (when working around heavy equipment).

11.14.1.1 Early Symptoms of Heat-Related Problems

- Decline in task performance;
- Lack of coordination;
- Decline in alertness;
- Unsteady walk;
- Excessive fatigue;
- Muscle cramps; and
- Dizziness.

11.14.1.2 Heat Stress Disorders

Refer to **Section 11.2.12**, Heat Stress Illness / Cold Stress Injury First Aid for symptoms and treatment of heat related illnesses.

11.14.1.2.1 Preventive Measures

Proper training and preventive measures will help avert serious illness and loss of work productivity. Preventing heat stress is particularly important because once someone suffers from heat exhaustion, that person may become predisposed to additional heat injuries. In order to avoid heat-related illnesses, proper preventive measures will be implemented whenever the temperature reaches 70°F. These preventive measures represent the minimal steps to be taken and include the following procedures:

The SSHO or other authorized person will observe each site worker prior to the start of daily operations, and periodically throughout the day, to determine the individuals susceptible to heat induced stress. Evidence of extreme dehydration, illness, effects from prescription or residual impacts of off-duty alcohol use may require the SSHO to restrict the worker's activities until such time as the worker is fit for duty. Personnel identified as being at high risk for heat stress who are allowed to participate in site operations will be monitored frequently by the SSHO.

Site workers will be trained to recognize and treat heat-related illnesses. This training will include the signs, symptoms and treatment of heat stress disorders as outlined in this section of the Site Safety and Health Plan (SSHP) (**Attachment A**).

To maintain workers' body fluids at normal levels, workers will be encouraged to drink a minimum of approximately sixteen ounces of liquids prior to start of work in the morning, after lunch and prior to leaving the site at the conclusion of the day's activities. Disposable four- to twelve-ounce cups and liquids will be provided on-site. Liquids containing caffeine should be avoided.

When ambient conditions and site workload requirements dictate, as determined by the SSHO, workers will be required to drink a minimum of 16 to 32 ounces of liquids during each rest cycle. The normal thirst mechanism is not sensitive enough to ensure enough water will be consumed to replace lost sweat. When heavy sweating occurs, workers shall be encouraged to drink even

though they may not be thirsty. A shelter or shaded area may be provided where workers are protected from direct sunlight during rest periods.

Monitoring of ambient or physiological heat stress indices will be conducted to allow prevention and/or early detection of heat induced stress. Monitoring will be conducted in accordance with applicable paragraphs of this SSHP.

Site workers will be given time to acclimatize to site work conditions, temperature, protective equipment, and workload. Acclimatization is the adaptive process that usually takes two to six days of continued work in hot environments, resulting in a decrease of the physiological strain and allowing the worker's body to adjust to the level and type of work required by the application of a constant environmental stress.

This process involves a gradual increase in the individual's workload over the required period, the length of which depends upon the nature of the work performed, ambient temperatures, and the individual's susceptibility to heat stress.

Work schedules will be adjusted as follows:

- Modify work/rest schedules according to monitoring requirements;
- Mandate work slowdowns as needed;
- Rotate personnel: alternate job functions to minimize over-stress or overexertion at one task;
- Add additional personnel to work teams; and
- Perform work during cooler hours of the day if possible.

Workers will be encouraged to achieve and maintain an optimum level of physical fitness. Increased physical fitness allows workers to better tolerate and respond to hot environments and heavy workloads. In comparison to an unfit person, a fit person will have: less physiological strain, a lower heart rate and body temperature and a more efficient sweating mechanism. Alcohol should not be consumed in a hot environment because the loss of body fluids increases the risk of heat stress.

11.14.1.3 Heat Stress Monitoring

Because the incidence of heat stress depends on a variety of factors, all workers shall be monitored. Initially, the frequency of physiological monitoring depends on the air temperature adjusted for solar radiation and the level of physical work. The length of the work cycle will be governed by the frequency of the required physiological monitoring. Physiological monitoring is the preferred method of heat stress control.

Monitoring of personnel wearing PPE may begin when the ambient temperature is 72°F or above in accordance with **Table 11-11**. Monitoring frequency should increase as the ambient temperature increases or as slow recovery rates are observed.

A person with a current first aid certification who is trained to recognize heat stress symptoms will perform heat stress monitoring.

When monitoring the worker physically, measure:

- Heart rate. Count the radial pulse during a 30-second period as early as possible in the rest period;
- If the heart rate exceeds 110 beats per minute (bpm) at the beginning of the rest period, shorten the next work cycle by one-third and keep the rest period the same;

- If the heart rate still exceeds 110 bpm at the next rest period, shorten the following work cycle by one-third;
- Oral temperature. Use a clinical thermometer (three minutes under the tongue) or similar device to measure the oral temperature at the end of the work period (before drinking);
- If oral temperature exceeds 99.6°F (37.6 degrees Centigrade [°C]), shorten the next work cycle by one-third without changing the rest period;
- If oral temperature still exceeds 99.6°F (37.6°C) at the beginning of the next rest period, shorten the following cycle by one-third; and
- Do not permit a worker to wear a semi-impermeable or impermeable garment when oral temperature exceeds 100.6°F (38.1°C).

Table 11-11 Physiological Monitoring for Fit and Acclimatized Workers

Adjusted Temperature	Normal Work Ensemble	Impermeable Ensemble
90°F (32.2°C) or above	After each 45 min. of work	After each 15 min. of work
87.5°-90°F(30.8°-32.2°C)	After each 60 min. of work	After each 30 min. of work
82.5°-87.5°F (28.1°-28.1°C)	After each 90 min. of work	After each 60 min. of work
77.5°-82.5°F (25.3°-28.1°C)	After each 120 min. of work	After each 90 min. of work
72.5°-77.5°F (22.5°-25.3°C)	After each 150 min. of work	After each 120 min. of work

a. For work levels of 250 kilocalories/hour
b. Calculate the adjusted air temperature by using this equation: adjusted air temperature (°F) = air temperature (°F) + (13 x % sunshine). Measure air temperature with a standard mercury-in-glass thermometer, with the bulb shielded from radiant heat. Estimate percent sunshine by judging what percent time the sun is not covered by clouds that are thick enough to produce a shadow. (100 percent sunshine = no cloud cover and a sharp, distinct shadow; 0 percent sunshine = no shadows.)
c. A normal work ensemble consists of cotton coveralls or other cotton clothing with long sleeves and pants.

11.14.1.4 Wet Bulb, Dry Globe Temperature Monitoring

Other methods for determining heat stress monitoring, such as the wet bulb globe temperature (WBGT) index from American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Value (TLV) booklet or portable heat stress monitoring instrumentation can be used. When workers are wearing permeable clothing (i.e. standard cotton work clothes), follow recommendations for monitoring requirements and suggested work/rest schedules in the current ACGIH TLV for Heat Stress.

For site conditions where personnel are working in Level D PPE, and the ambient temperature is greater than 72°F, the SSHO may conduct WBGT monitoring to assist in controlling the potential for site workers experiencing heat-related adverse health effects. The SSHO may take readings on a WBGT monitor throughout the day to determine the work/rest schedule to be implemented.

The values outlined in **Table 11-11** and **Table 11-12** are designed such that nearly all acclimatized, fully clothed workers with adequate water and electrolyte replacement liquids intake will be able to function without the body temperature exceeding 100.4°F (38°C).

Table 11-12 Permissible WBGT Heat Exposure TLVs

Work – Rest Regimen	WORK LOAD		
	Light	Moderate	Heavy
Continuous work	86 °F (30.0 °C)	80 °F (26.7 °C)	77 °F (25.0 °C)
75% Work - 25% Rest, each hour	87 °F (30.6 °C)	82 °F (28.0 °C)	78 °F (25.9 °C)
50% Work - 50% Rest, each hour	89 °F (31.4 °C)	85 °F (29.4 °C)	82 °F (27.9 °C)
25% Work - 75% Rest, each hour	90 °F (32.2 °C)	88 °F (31.1 °C)	86 °F (30.0 °C)

*Consult the ACGIH TLV booklet for definitions of Light, Moderate and Heavy workloads. Values are given in °F and (°C) WBGT, and are intended for workers wearing single layer summer type clothing. Use of semi or totally impermeable clothing requires monitoring in accordance with the Heat Stress Prevention Program. As workload increases, the heat stress impact on a non-acclimated worker is exacerbated. For non-acclimated workers performing a moderate level of work, the permissible heat exposure TLV should be reduced by approximately 2.5°C.

11.14.1.5 Heat Stress Documentation

The SSHO is responsible for recording all heat stress-related information, including training sessions and monitoring data. Training sessions will be documented on the Training Acknowledgement Form, and WBGT data and other information will be recorded in the field notebook.

11.14.2 Cold Stress

If work on this project is conducted in the winter months, thermal injury due to cold exposure can become a problem for field personnel. Work will cease under unusually hazardous conditions (e.g. wind-chill less than 0°F, or wind-chill less than 10°F with precipitation).

Systemic cold exposure is referred to as hypothermia. Localized cold exposure is generally labeled frostbite. Recognition of the symptoms of cold-related illness will be discussed during the health and safety briefing conducted prior to the onset of site activities. Refer to the most recent revision of the ACGIH TLV for Chemical Substances and Physical Agents for additional information on cold stress prevention, monitoring, and work-warming regimens.

Refer to **Section 11.2.12**, Heat Stress Illness / Cold Stress Injury First Aid for symptoms and treatment of cold related illnesses.

11.14.2.1 Prevention of Cold-Related Illness

- Educate workers to recognize the symptoms of frostbite and hypothermia;
- Ensure the availability of an enclosed, heated environment. The nearest heated environment will be the interior of the vehicles at the site;
- Ensure the availability of dry changes of clothes;
- Record temperature readings; and
- Ensure the availability of warm beverages, preferably non-caffeinated.

11.14.2.2 Monitoring for Cold Exposure

Cold stress monitoring will be conducted in accordance with the ACGIH cold stress TLV. The TLV objective is to prevent the deep body core temperature from falling below 96.8°F and to prevent cold injury to body extremities. Temperature monitoring and recording will be initiated in the following situations:

- At the SSHO discretion based on changes in worker's performance or mental status;
- At the worker's request;

- As a screening measure whenever anyone worker on the site develops hypothermia; and
- Any person developing moderate hypothermia (a core temperature of 92°F) cannot return to work for 48 hours.

11.15 Crystalline Silica Monitoring Plan

Not applicable.

11.16 Night Operations Lighting Plan

Not applicable.

11.17 Fire Prevention Plan

Potential causes of fires include tobacco smoking, internal combustion engine powered equipment and the use of equipment that is not intrinsically safe while monitoring landfill gas. Observing the following precautions will prevent fires:

- Smoke only in designated areas;
- Ensure internal combustion equipment has proper exhaust systems with spark arresting devices;
- Do not park vehicles where vegetation may contact the exhaust system;
- Store flammable liquids at least 50 feet from potential ignition sources and in a well-ventilated area;
- Refuel equipment at least 50 feet from potential ignition sources; and
- Use intrinsically safe equipment when working in the vicinity near potentially explosive atmospheres.

At least one fire extinguisher, with a minimum rating of 2-A:10-B:C, will be kept on each vehicle to fight incipient fires. Shovels and other hand tools may also be used to fight small fires to prevent them from becoming wild land fires. Personnel must not endanger themselves when fighting fires and must notify the fire department even if the fire has been extinguished so firefighters can determine if a residual ignition hazard exists.

11.18 Wild Land Fire Management Plan

Personnel safety is paramount in the event of a wild land fire. The precautions in **Section 11.17** are applicable to the prevention of wild land fires. Personnel will report wild land fires by calling **911**. In the event of a wild land fire, all personnel will evacuate the work site. When safe to do so, any materials such as flammable liquids which have the potential to accelerate the fire will be removed from the site.

11.19 Hazardous Energy Control Plan

Not applicable.

11.20 Critical Lift Plan

Not applicable.

11.21 Contingency Plan for Severe Weather

Refer to **Section 11.7.6.14**, Weather-Related Hazards.

11.22 Float Plan

Not applicable.

11.23 Site Specific Fall Protection and Prevention Plan

Refer to **Section 11.7.6.1**, Work at Heights.

11.24 Demolition Plan

Not applicable.

11.25 Excavation and Trenching Plan

Site work will include the excavation of soil as necessary to meet the objectives of the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP). Soil confirmation samples may be collected from the bottom of excavations. Workers will not enter excavations greater than 4 feet in depth to collect soil samples. Excavation to depths greater than 5 feet is not anticipated. Excavations will be inspected daily and upon changing condition by the SSHO. Refer to safety precautions in **Section 11.7.6.3**, Excavation/Trenching.

11.26 Emergency Rescue (Tunneling)

Not applicable.

11.27 Underground Construction, Fire Prevention and Protection Plan

Not applicable.

11.28 Compressed Air Plan

Not applicable.

11.29 Formwork and Shoring Erection and Removal Plan

Not applicable.

11.30 Precast Concrete Plan

Not applicable.

11.31 Lift Slab Plans

Not applicable.

11.32 Steel Erection Plan

Not applicable.

11.33 Blasting Safety Plan

Not applicable.

11.34 Diving Plan

Not applicable.

11.35 Confined Space Plan

Not applicable.

11.36 Exposure Monitoring/Air Sampling Program

The SSHO or designee is responsible for completing exposure monitoring during field activities where there is potential exposure to combustible gases, oxygen deficiency, airborne dust, specific airborne contaminants, and physical agents above Immediately Dangerous to Life or Health (IDLH) levels, OSHA PELs, ACGIH TLVs, or other published exposure guidelines. The components of the exposure monitoring program and the frequency of such personal and environmental monitoring is established in this SSHP and is based on the work tasks to be completed, work methods to be used, contaminants and concentrations present.

11.36.1 Monitoring Program Objectives

The primary objectives of air monitoring programs implemented at the site are to identify and quantify airborne contaminant concentrations and monitor physical hazards during site work (e.g. carbon monoxide, airborne dust, noise, heat and cold stress). The data obtained is used to help establish criteria for use of engineering controls and safe work practices, upgrade/downgrade of PPE, work stoppage or emergency evacuation, and prevention or minimization of public exposures.

11.36.2 Exposure Monitoring Plan

Monitoring is conducted to determine personnel exposures to chemical contaminants and physical agents during various site activities. Monitoring during project operations are completed by the SSHO.

Monitoring will be conducted on those site personnel who are likely to have the highest exposures to hazardous substances and health hazards likely to be present above 50 percent of the permissible exposure limits (PELs) or TLV (lowest value). If the site personnel likely to have the highest exposures have exposures over PELs or TLVs, then monitoring will be continued and expanded to establish all employees likely to have exposures above those limits.

The results of air monitoring will be recorded or attached onto the daily safety inspection log maintained by the SSHO. The SSHO will provide exposure monitoring results for specific contaminant monitoring to individuals monitored within five days of receipt of results. The SSHO will maintain copies of such exposure monitoring records at the site for the duration of the project. Upon completion of the project, the exposure monitoring records will be maintained similarly to medical records and placed in each applicable employee's exposure monitoring record files for the duration of employment plus 30 years.

11.36.3 Direct-Reading (Real-time) Instrument Air Monitoring

11.36.3.1 Airborne Dust

Airborne dust monitoring will occur when intrusive activities are conducted. Monitoring will be conducted at the site perimeter (of each discrete intrusive activity site) to verify no off-site migration of airborne dust (and entrained solid contaminants) occurs during intrusive activities. Monitoring is also conducted to measure worker exposures during intrusive work activities. An airborne dust action level will be used to minimize worker exposures and ensure that no off-site migration of contaminants occurs by triggering use of additional dust control measures if the action level is exceeded.

A personal data-logging Real-time Aerosol Monitor (RAM) will be used during dust-generating activities to measure airborne dust. The RAM is a direct reading airborne dust monitor that is capable of measuring dust with a particle size of 0.1-10 micrometer (µm) over a concentration range of 0.001-100 milligrams per cubic meter (mg/m^3). The instrument can provide instantaneous data as well as time-weighted average (TWA) information. The instrument will be zeroed before use with a Z-Bag unit per manufacturer instructions.

11.36.3.2 Photoionization Detector

VOC monitoring will occur at the beginning of all site activities, and continuously during intrusive work. A VOC action limit will be used to minimize worker exposures during site work. If the VOC concentrations exceed the action limit; site workers will don appropriate respiratory protection.

A Photoionization Detector (PID) will be used to evaluate VOC concentrations in the breathing zone of personnel. The PID is equipped with a 10.6 electron Volt (eV) UV lamp. The PID is to be calibrated prior to use in a clean, non-hazardous area to a known concentration (100 ppm) of isobutylene. Verification of appropriate instrument response will be performed after initial calibration.

11.36.3.3 Landfill Gas Monitor

A landfill gas monitor is used for sampling and analysis of gas composition. The unit will provide instantaneous concentrations in percent by volume of CH_4, CO_2, Oxygen (O_2), LEL, and balance gas. Units require field calibration to a known multi-gas mixture. Units will be certified intrinsically safe for landfill use and the verification of appropriate instrument response will be performed after initial calibration.

11.36.4 Time-Integrated Air Monitoring

The RAM may also be used to measure eight-hour TWA exposures to respirable dust. The RAM has a detection limit of 0.001 mg/m^3. The SHM and CIH will evaluate the need for personnel exposure monitoring at individual sites, in consultation with the SSHO, and based on initial real-time monitoring results. Monitoring will be conducted based on initial real-time monitoring dust monitoring and soil concentrations to determine personnel exposure during intrusive activities.

12.0 SITE SAFETY AND HEALTH PLAN

The following information which contains site-specific details is contained in **Attachment A** to this plan:

- Site Description;
- Contamination Characterization;
- AHA;
- Staff Organization, Qualifications, and Responsibilities;
- Training;
- Medical Surveillance;
- Exposure Monitoring/Air Sampling Program;
- PPE;
- Heat and Cold Stress;
- SOPs, Engineering Controls, and Work Practices;
- Site Control Measures;
- On-Site Communications;
- Personal Hygiene and Decontamination;
- Emergency and First Aid Equipment; and
- ERP.

Appendix 1

Training Acknowledgement Form

Training Acknowledgement Form

Project: Performance-Based Restoration - W9128F-10-D-0025, DO #0002

Location: Joint Base Andrews Naval Air Facility Washington

Employee/Visitor Name: _____

The contract for the above project requires the following:

that you are provided with and complete formal and site-specific training;

that you are supplied with proper personal protective equipment (PPE) including respirators;

that you are trained in its use; and

that you receive a medical examination to evaluate your physical capacity to perform your assigned work tasks, under the environmental conditions expected, while wearing the required PPE (including respiratory protection).

Those things are done at no cost to you. By signing this certificate, you are acknowledging that your employer has met these obligations to you.

I HAVE READ, UNDERSTAND AND AGREE TO FOLLOW THE ACCIDENT PREVENTION PLAN, SITE SAFETY AND HEALTH PLAN AND ACTIVITY HAZARD ANALYSES FOR THIS SITE.

_____ _____

Name Date

FORMAL TRAINING: I have completed the following training courses that meet OSHA requirements:

	Date Completed
40-hour HAZWOPER	_____
8-hour supervisory	_____
8-hour refresher	_____

(You must provide copies of course completion certificates for these courses.)

SITE-SPECIFIC TRAINING: I have been provided and have completed the site-specific training required by this Contract. _____ (Initials)

RESPIRATORY PROTECTION: I have been trained and medically monitored in accordance with the criteria in my employer's respiratory protection program. I have been trained in the proper work procedures and use and limitation of the respirator I will wear. I have been trained in and will abide by the facial hair policy. _____ (Initials) or _____ Not applicable.

RESPIRATOR FIT-TEST TRAINING: I have been trained in the proper selection, fit, use, care, cleaning, maintenance, and storage of the respirator that I will wear. I have been fit-tested in accordance with the criteria in the corporate Respiratory Program and have received a satisfactory fit. I have been taught how to properly perform positive and negative pressure fit-checks upon donning negative pressure respirators each time. _____ (Initials) or _____ Not applicable.

MEDICAL EXAMINATION: I have had a medical examination within the last 12 / 24 (circle one) months which was paid for by my employer. The examination met the requirements of 29 CFR 1910.120 and 29 CFR 1910.134. A physician made a determination regarding my physical capacity to perform work tasks on the project while wearing protective equipment, possibly including a respirator. I was personally provided a copy and informed of the results of that examination. The physician determined that there:

_____Were no limitations to performing the required tasks.

_____Were identified physical limitations to performing the required work tasks.

Date of medical examination: _____

Employee/Visitor Signature:	
Printed Name:	
Date:	
Last 4 Numbers of Social Security Number:	XXX-XX-

Appendix 2

Safety and Health Meeting Report Form

Bay West
Customer-Focused Environmental & Industrial Solutions

Safety and Health Meeting Report

General Information			
Date / Time		Job Number	J
Client / Location			
Site Safety and Health Officer		Duration	Minutes
Type of Meeting	☐ Daily ☐ Weekly ☐ Monthly ☐ Other:		

Items / Issues Discussed	
1.	
2.	
3.	
4.	
5.	
6.	
7.	

	Safety Concerns / Issues Raised	Action Items / Follow-up Needed
1.		
2.		
3.		
4.		
5.		
6.		
7.		

Comments / Remarks

Forward a copy of this completed Safety and Health meeting Report to the Safety and Health Manager. Attach a copy of meeting minutes (if applicable).

Bay West
Customer-Focused Environmental & Industrial Solutions

Safety and Health Meeting Report

Safety and Health Meeting Attendees		
Printed Name	**Signature**	**Company**

Appendix 3

Inspection Forms

Bay West Daily Equipment Maintenance Log

Date:			Job Number:			
Time (hrs)	**EQ Group**	**TW #**	**Description**	**Parts**	**Part #**	**Cost**

DAILY SAFETY INSPECTION CHECKLIST

Date:	Inspector:	Location:

Weather conditions:

	N/A	Yes	No
1. Site Conditions			
Is site kept clean and orderly?			
Are break areas in a sanitary condition?			
Are work zones marked and entry enforced?			
Is the CRZ established, stocked with decon materials, and in order?			
Are radios provided for communication?			
Are slipping and tripping hazards identified and marked or removed?			
Is the site control log used and maintained?			
2. Emergency Equipment			
Are fire extinguishers provided and in good condition?			
Are first aid kits provided and stocked?			
Have SCBAs been inspected?			
Are site telephones posted with emergency information?			
3. Heavy Equipment Use			
Is equipment inspected daily?			
Are workers restricted from working under suspended loads?			
Is equipment clear of power lines?			
4. Excavations and Trenches			
Have utilities been marked?			
Are excavations inspected on daily basis?			
Are excavations shored?			
Is egress provided?			
5. Personal Protective Equipment			
Is PPE used as required?			
Is PPE maintained, cleaned, and stored properly?			
Are adequate stocks of PPE available?			
6. Electrical			
Are GFCIs used on extension cords?			
Are extension cords properly used and in good condition?			
7. Work Practices			
Is buddy system used?			
Is eating, drinking or tobacco used only in approved areas?			
Is decontamination performed as required (personnel and equipment)?			
8. Other Items:			

Provide documentation of deficiencies (items marked ‘no’) on the back of this form.

DEFICIENT ITEMS OR ACTIVITIES IDENTIFIED		
Item No.	**Description of Deficiency**	**Corrective Action Performed/Planned**

EXCAVATION INSPECTION FORM

Complete this form at the beginning of each shift or after an event that may have impacted the safety of an excavation.

Job No.:	Competent Person:
Date of Inspection:	Time of Inspection: a.m./p.m.
Description of Excavation:	
Excavation activities performed/planned:	

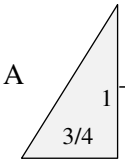
Soil type and protective system:

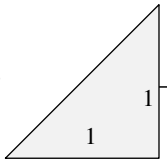
____ Sloping or Benching

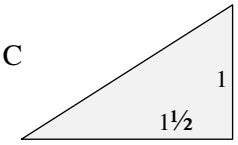
____ Stable Rock _____ Type A _____ Type B ___ Type C

(Vertical)

____ Shoring/shielding Utilized?







INSPECTION CHECKLIST	N/A	YES	NO
All Excavations:			
Is a barrier provided to identify and guard the excavation boundary?			
Have underground utilities been identified and marked?			
Is clearance (10 ft minimum) or guarding provided to overhead power lines?			
Is excavated material and equipment placed at least 2' from the excavation?			
Do ground workers wear safety vests when working near heavy equipment?			
Are adjacent structures properly stabilized if impacted by the excavation?			
Is there evidence of water that may impact the safety of the excavation?			
Excavations >4 feet deep			
Is a means of egress provided no more than 25' from the work?			
Is there a potential for a hazardous atmosphere in the excavation?			
If yes, is the atmosphere monitored and emergency equipment provided?			
Excavations >5 feet deep			
Is a cave-in protective system employed?			
Is sloping at the proper angle?			
Is shoring or shielding properly designed, constructed, installed and maintained?			
Excavation >20 feet deep			
Has a Registered Professional Engineer designed the protective system?			
Are the designs followed?			

Competent Person Signature:_____

File this completed checklist with the project file.

SAFETY INSPECTION CHECKLIST FOR CONSTRUCTION EQUIPMENT

(Including Cranes, Derricks, and Hoisting Equipment)

PROJECT:	CONTRACTOR:	CONTRACT NO.:
TYPE AND MAKE OF EQUIPMENT:	MODEL:	SERIAL NO.:

Before any machinery or mechanized equipment is placed in sue, it shall be inspected and tested by a competent mechanic and certified to be in good operating condition. Records of tests and inspections shall be maintained as part of the active contract file at Project or Resident Office. Checklist set forth herein requires the application of EM 385-1-1, Safety and Health Requirements Manual.

CHECKLIST	**Yes**	**No**	**N/A**
1. Are adequate and serviceable fire extinguishers provided?			
2. Are all wire rope cables in good condition?			
3. Are wire rope, sockets, splices, thimbles and clips adequate and properly attached?			
4. Are hooks, safety nooks, shackles, rings, etc., in good condition?			
5. Are necessary platforms, footwalks, etc., provided?			
6. Are access steps, platforms, etc., provided with non-slip surfaces?			
7. Is operator protected against the elements, falling or flying objects, swinging loads, and similar hazards?			
8. Are all glasses in operator's compartment safety glass and in good repair?			
9. Is suitable access provided to lubrication points?			
10. Do all modifications, extensions, replacement parts, and/or repairs to equipment maintain the same factor of safety as original designed equipment?			
11. Are drums for load lines equipped with at least one positive holding device, applied directly to the motor shaft or some part of the train gear?			
12. Is there sufficient cable to allow two full wraps of cable on drums at all working positions?			
13. Are adequate headlights, taillights and turn signals provided and are they in proper operating condition?			
14. Are all approved brakes on wheeled equipment and in good operating condition?			
15. Do windshields have wipers in proper operating condition?			
16. Are rear view mirrors provided?			
17. Are operating levers equipped with latch or other devices to prevent accidental starting?			

CHECKLIST	Yes	No	N/A
18. Is engine equipped with power-operated starting device in operative condition?			
19. Do all pressure vessels have valid inspection certificates?			
20. Are reverse signal alarms on equipment?			
21. Are belts, gears, shafts, electrical contacts, etc., adequately guarded?			
22. Are all hot pipes and surfaces suitably guarded?			
23. Are fuel tanks located so that spills or overflows will not come in contact with engine or exhaust?			
24. Are exhausts and discharges so directed as not to endanger workmen or obstruct view of operator?			
25. Are guards in place on equipment with drop type skip pans?			
26. Are adequate seats provided for all riders?			
27. Are tires in serviceable condition? Are testing/inspections documented?			
28. Is steering linkage and tie rod in good operating condition? Are testing/inspections documented?			
29. Are dump bodies provided with holding device or other suitable device for locking body in raised position?			
30. Are tailgate dumping devices so arranged that operator will be in the clear while dumping loads?			
31. Are trip-handles provided on tailgates to facilitate handling?			
32. Is air hose free from leaks or defects?			
33. Are safety lashings for quick make-up type connections provided?			
34. Is acceptable spark arrestor installed and working?			
35. Do heating devices comply with references?			
36. Does welding equipment comply with code requirements?			
37. Is equipment adequately grounded?			
38. Do electrical components comply with code?			
39. Are required pressure, temperature, or relief gages and valves installed and operable?			
40. Are approved seat belts and rollover protection provided?			
41. Is recommended preventive maintenance being followed?			
42. Do helicopter cranes meet construction requirements?			

CHECKLIST	Yes	No	N/A
43. Do hydraulic jacks meet special safety conditions?			
44. Is concrete equipment fitted with adequate safety devices?			
45. Are elevating and rotating work platforms in conformance with ANSI A92.2?			
46. Do conveyors, cableways, and related equipment conform to ANSI B20.0?			
47. Are pile drivers equipped with all appropriate safety devices?			
48. Do material hoists conform to ANSI A10.5?			
49. Do passenger elevators conform to ANSI A10.4? Do temporary hoists in accordance to ANSI A20.22?			
50. Do hand and power tools comply with applicable ANSI standards?			
The following six questions apply to Cranes and Hoisting Equipment only and need not be answered for other construction equipment.			
51. Is high voltage sign posted?			
52. Is equipment fitted with positive stops for rotation when near power lines?			
53. Is there any visible evidence to damage to boom?			
54. Is the boom position indicator operating and visible to operator?			
55. Have all operators had a current physical examination?			
56. Is braking equipment capable of effectively braking, lowering and safety holding a load of at least the full rated load as required?			

REMARKS:

CERTIFICATION: *I hereby certify that this item of equipment is in good operating condition and that it meets all above requirements except as noted under remarks.*

_____ _____

Signature of Competent Mechanic Date

_____ _____

Signature of Superintendent/Quality Control Engineer Date

Appendix 4

USACE Accident Investigation and Reporting Form

(For Safety Staff only)	REPORT NO.	EROC CODE	**UNITED STATES ARMY CORPS OF ENGINEERS ACCIDENT INVESTIGATION REPORT** *(For Use of this Form See Help Menu and USACE Suppl to AR 385-40)*	**REQUIREMENT CONTROL SYMBOL: CEEC-S-8(R2)**

1. **ACCIDENT CLASSIFICATION**

PERSONNEL CLASSIFICATION	INJURY/ILLNESS/FATAL	PROPERTY DAMAGE	MOTOR VEHICLE INVOLVED	DIVING
GOVERNMENT ☐ CIVILIAN ☐ MILITARY	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ CONTRACTOR	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ PUBLIC	☐ FATAL ☐ OTHER		☐	

2. **PERSONAL DATA**

a. Name *(Last, First, MI)*	b. AGE	c. SEX ☐ MALE ☐ FEMALE	d. SOCIAL SECURITY NUMBER	e. GRADE

f. JOB SERIES/TITLE	g. DUTY STATUS AT TIME OF ACCIDENT	h. EMPLOYMENT STATUS AT TIME OF ACCIDENT
	☐ ON DUTY ☐ TDY ☐ OFF DUTY	☐ ARMY ACTIVE ☐ ARMY RESERVE ☐ VOLUNTEER ☐ PERMANENT ☐ FOREIGN NATIONAL ☐ SEASONAL ☐ TEMPORARY ☐ STUDENT ☐ OTHER *(Specify)*

3. **GENERAL INFORMATION**

a. DATE OF ACCIDENT *(month/day/year)*	b. TIME OF ACCIDENT *(Military time)* hrs	c. EXACT LOCATION OF ACCIDENT	d. CONTRACTOR'S NAME (1) PRIME: (2) SUBCONTRACTOR:

e. CONTRACT NUMBER	f. TYPE OF CONTRACT	g. HAZARDOUS/TOXIC WASTE ACTIVITY
☐ CIVIL WORKS ☐ MILITARY ☐ OTHER *(Specify)*	☐ CONSTRUCTION ☐ SERVICE ☐ A/E ☐ DREDGE ☐ OTHER *(Specify)*	☐ SUPERFUND ☐ DERP ☐ IRP ☐ OTHER *(Specify)*

4. **CONSTRUCTION ACTIVITIES ONLY** *(Fill in line and corresponding code number in box from list - see help menu)*

a. CONSTRUCTION ACTIVITY	(CODE) #	b. TYPE OF CONSTRUCTION EQUIPMENT	(CODE) #

5. INJURY/ILLNESS INFORMATION *(Include name on line and corresponding code number in box for items e, f & g - see help menu)*

a. SEVERITY OF ILLNESS/INJURY	(CODE) #	b. ESTIMATED DAYS LOST	c. ESTIMATED DAYS HOSPITALIZED	d. ESTIMATED DAYS RESTRICTED DUTY

e. BODY PART AFFECTED	(CODE) #	g. TYPE AND SOURCE OF INJURY/ILLNESS	(CODE) #
PRIMARY		TYPE	
SECONDARY		SOURCE	
f. NATURE OF ILLNESS/INJURY			

6. PUBLIC FATALITY *(Fill in line and correspondence code number in box - see help menu)*

a. ACTIVITY AT TIME OF ACCIDENT	(CODE) #	b. PERSONAL FLOATATION DEVICE USED?
		☐ YES ☐ NO ☐ N/A

7. MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE	b. TYPE OF COLLISION	c. SEAT BELTS	USED	NOT USED	NOT AVAILABLE
☐☐ PICKUP/VAN ☐☐ AUTOMOBILE ☐☐ TRUCK ☐☐ OTHER *(Specify)*	☐ SIDE SWIPE ☐ HEAD ON ☐ REAR END ☐ BROADSIDE ☐ ROLL OVER ☐ BACKING ☐ OTHER *(Specify)*	(1) FRONT SEAT			
		(2) REAR SEAT			

8. PROPERTY/MATERIAL INVOLVED

a. NAME OF ITEM	b. OWNERSHIP	c. $ AMOUNT OF DAMAGE
(1)		
(2)		
(3)		

9. VESSEL/FLOATING PLANT ACCIDENT *(Fill in line and correspondence code number in box from list - see help menu)*

a. TYPE OF VESSEL/FLOATING PLANT	(CODE) #	b. TYPE OF COLLISION/MISHAP	(CODE) #

10. ACCIDENT DESCRIPTION *(Use additional paper, if necessary)*

11. **CAUSAL FACTOR(S)** *(Read Instruction Before Completing)*

a. (Explain YES answers in item 13)	YES	NO
DESIGN: Was design of facility, workplace or equipment a factor?	☐	☐
INSPECTION/MAINTENANCE: Were inspection & maintenance procedures a factor?	☐	☐
PERSON'S PHYSICAL CONDITION: In your opinion, was the physical condition of the person a factor?	☐	☐
OPERATING PROCEDURES: Were operating procedures a factor?	☐	☐
JOB PRACTICES: Were any job safety/health practices not followed when the accident occurred?	☐	☐
HUMAN FACTORS: Did any human factors such as, size or strength of person, etc., contribute to accident?	☐	☐
ENVIRONMENTAL FACTORS: Did heat, cold, dust, sun, glare, etc., contribute to the accident?	☐	☐

a. *(CONTINUED)*	YES	NO
CHEMICAL AND PHYSICAL AGENT FACTORS: Did exposure to chemical agents, such as dust, fumes, mists, vapors or physical agents, such as, noise, radiation, etc., contribute to accident?	☐	☐
OFFICE FACTORS: Did office setting such as, lifting office furniture, carrying, stooping, etc., contribute to the accident?	☐	☐
SUPPORT FACTORS: Were inappropriate tools/resources provided to properly perform the activity/task?	☐	☐
PERSONAL PROTECTIVE EQUIPMENT: Did the improper selection, use or maintenance of personal protective equipment contribute to the accident?	☐	☐
DRUGS/ALCOHOL: In your opinion, was drugs or alcohol a factor to the accident	☐	☐

b. WAS A WRITTEN JOB/ACTIVITY HAZARD ANALYSIS COMPLETED FOR TASK BEING PERFORMED AT TIME OF ACCIDENT?

☐ YES *(If yes, attach a copy.)* ☐ NO

12. **TRAINING**

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK?	b. TYPE OF TRAINING.	c. DATE OF MOST RECENT FORMAL TRAINING.
☐ YES ☐ NO	☐ CLASSROOM ☐ ON JOB	(Month) (Day) (Year)

13. **FULLY EXPLAIN WHAT ALLOWED OR CAUSED THE ACCIDENT; INCLUDE DIRECT AND INDIRECT CAUSES** *(See instruction for definition of direct and indirect causes.) (Use additional paper, if necessary)*

a. DIRECT CAUSE

b. INDIRECT CAUSE(S)

14. **ACTION(S) TAKEN, ANTICIPATED OR RECOMMENDED TO ELIMINATE CAUSE(S).**

DESCRIBE FULLY:

15. **DATES FOR ACTIONS IDENTIFIED IN BLOCK 14.**

a. BEGINNING (Month/Day/Year)	b. ANTICIPATED COMPLETION (Month/Day/Year)		
c. SIGNATURE AND TITLE OF SUPERVISOR COMPLETING REPORT CORPS _____ CONTRACTOR _____	d. DATE *(Mo/Da/Yr)*	e. ORGANIZATION IDENTIFIER *(Div, Br, Sect)*	f. OFFICE SYMBOL

16. **MANAGEMENT REVIEW** *(1st)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

17. **MANAGEMENT REVIEW** *(2nd - Chief Operations, Construction, Engineering, etc.)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE	TITLE	DATE

18. **SAFETY AND OCCUPATIONAL HEALTH OFFICE REVIEW**

a. ☐ CONCUR b. ☐ NON CONCUR c. ADDITIONAL ACTIONS/COMMENTS

SIGNATURE	TITLE	DATE

19. **COMMAND APPROVAL**

COMMENTS

COMMANDER SIGNATURE	DATE

*U.S. GOVERNMENT PRINTING OFFICE: 1993-0-791-757

10. ACCIDENT DESCRIPTION ***(Continuation)***

13a. DIRECT CAUSE ***(Continuation)***

13b. INDIRECT CAUSES *(Continuation)*

14. ACTION(S) TAKEN, ANTICIPATED, OR RECOMMENDED TO ELIMINATE CAUSE(S) *(Continuation)*

Appendix 5

Incident Investigation Report
First Report of Injury
Near Miss Report

Bay West
Customer-Focused Environmental & Industrial Solutions

Incident Investigation Report

Instructions: The Site Supervisor is to notify the Project Manager, Safety and Health Manager (SHM) and Team Leader as soon as practical after the incident. The employee is to complete this form and submit it to the SHM no later than **48 hours** after the incident. The SHM will be responsible for notifying each individual listed below. In the absence of the SHM, the Project Manager will be responsible for routing this form. Questions regarding this form and/or the incident reporting procedure can be directed to the SHM.

	Comments	Initial
Project Manager		
HR		
Shop Manager		
CFO		
Team Leader		
CIH (federal projects)		
Health and Safety	OSHA recordable case? ☐Yes ☐No	

Section 1 – General Incident Information

Date of Report:		Time of Report:	
Person completing this form:		Signature:	
Person(s) involved:			
Witnesses:			
Project Manager:		BW Job #	J
Date of Incident:		Time of Incident:	

Where did the incident occur?	
What happened? – Describe the incident:	
Why did the incident occur? (In your opinion):	
What could have been done to prevent the incident?	
Have unsafe conditions been corrected?	☐Yes ☐No
If yes, what has been done?	
If no, what needs to be done?	
Root Cause Analysis (List underlining cause, i.e. lack of training, poor communication):	

Section 2 – Equipment Damage					
Description of equipment damage:					
Toolwatch #		Cost to replace/repair?		Bay West owned ☐ or rented ☐?	

Section 3 – Vehicle Damage					
REQUIRED: Attach Zurich Drivers Accident Report	☐	Attach Police Report		☐	
Name of Driver		Driver's License #		State:	
Description of vehicle damage:					
Toolwatch #		Vehicle Plate #			
Cost to replace/repair?		Bay West owned ☐ or rented ☐?			

Section 4 – Personal Injury or Illness	
Complete **First Report of Injury** form and attach (DMS# 112745)	☐
Treatment received:	

Section 5 – Incident Schematic / Notes
Provide visual aids or additional notes for the persons involved in the incident investigation. Use photographs, sketches, or graphics to document the location of the incident and how the incident occurred.

Minnesota Department of Labor and Industry
Workers' Compensation Division
PO Box 64221
St. Paul, MN 55164-0221
(651) 284-5030

First Report of Injury

See Instructions on Reverse Side
PRINT IN INK or TYPE
Enter dates in MM/DD/YYYY format.

Reset

FR01

DO NOT USE THIS SPACE

1. EMPLOYEE SOCIAL SECURITY #	2. OSHA Case #

3. DATE OF CLAIMED INJURY	4. Time of injury ☐ am ☐ pm	5. Time employee began work on date of injury ☐ am ☐ pm

6. EMPLOYEE Name (last, first, middle)	7. Gender ☐ M ☐ F	8. Marital Status ☐ Married ☐ Unmarried

9. Home Address	10. Home phone #	11. Date of birth

City	State	Zip Code	12. Occupation	13. Regular department	14. Date hired

15. Average weekly wage	16. Rate per hour	17. Hours per day	18. Days per week	19. Employment Status ☐ Full time ☐ Part time ☐ Seasonal ☐ Volunteer

20. Weekly value of:	Meals	Lodging	2nd Income	21. Apprentice ☐ Yes ☐ No

22. **Tell us how the injury occurred and what the employee was doing before the incident (give details).** *Examples: "Worker was driving lift truck with a pallet of boxes when the truck tipped, pinning worker's left leg under drive shaft." "Worker developed soreness in left wrist over time from daily computer key entry."*

23. **What was the injury or illness (include the part(s) of body)?** *Examples: chemical burn left hand, broken left leg, carpal tunnel syndrome in left wrist.*	24. **What tools, equipment, machines, objects, or substances were involved?** *Examples: chlorine, hand sprayer, pallet lift truck, computer keyboard.*

25. Did injury occur on employer's premises? ☐ Yes ☐ No If no, indicate name and address of place of occurrence	26. Date of first day of any lost time	27. Employer paid for lost time on day of injury (DOI) ☐ Yes ☐ No ☐ No lost time on DOI
	28. Date employer notified of injury	29. Date employer notified of lost time
	30. Return to work date	31. Date of death

32. TREATING PHYSICIAN (name, address, and phone)	33. HOSPITAL/CLINIC (name and address) (if any)	34. Emergency Room Visit ☐ Yes ☐ No 35. Overnight in-patient ☐ Yes ☐ No

36. **EMPLOYER** Legal name	37. EMPLOYER DBA name (if different)	
BAY WEST, INC.		
38. **Mailing** address	39. Employer FEIN	40. Unemployment ID#
5 EMPIRE DRIVE	41-1234511	
City / State / Zip Code	41. Employer's contact name and phone #	
SAINT PAUL MN 55103	ALYSSA REBISCHKE	
42. **Physical** address (if different)	43. Witness (name and phone)	
SAME		
City / State / Zip Code	44. NAICS code	45. Date form completed
	562910	

46. **INSURER** name	51. **CLAIMS ADMIN COMPANY** (CA) name (check one) ☐ Insurer ☐ TPA	
ZURICH AMERICAN INSURANCE CO.	ZURICH AMERICAN INSURANCE CO.	
47. Insured legal name	52. CA address	
BAY WEST, INC.	PO BOX 20048	
48. Policy # or self-insured certificate #	City / State / Zip Code	
WC 9375443	KANSAS CITY MO 64195	
49. Insurer FEIN	50. Date insurer received notice	53. CA FEIN / 54. Claim #
364233459		364233549 /

Copies to: Insurer, Employer, Employee, and Workers' Compensation Division (if no insurer)

Minnesota workers' compensation system employee information sheet

What does workers' compensation pay for?

- Medical care for the work injury, as long as it is reasonable and necessary
- Wage-loss benefits for part of your lost income (there is a three-calendar-day waiting period before these benefits start)
- Benefits for permanent damage or loss of function of a body part
- Benefits to your spouse and/or dependents if you die of a work injury
- Vocational rehabilitation services if you cannot return to your pre-injury job or to your pre-injury employer

How are workers' compensation benefits paid?

Your workers' compensation benefits are paid by an insurance company or your employer, if your employer is self-insured. State law sets the benefit levels. Please note: pursuant to statute, the insurer can obtain medical information specific to your work injury without your authorization.

If the insurer accepts your claim for wage loss benefits and you have been disabled for more than three calendar days:

- The insurer will send you a copy of the *Notice of Insurer's Primary Liability Determination* form stating your claim is accepted.

- The insurer must start paying wage-loss benefits within 14 days of the date your employer knows about your work injury and lost wages. The insurer must pay benefits on time. Wage-loss benefits are paid at the same intervals as your work paychecks.

If the insurer denies your claim for wage loss benefits:

- The insurer will send you a copy of the *Notice of Insurer's Primary Liability Determination* form stating it is denying primary liability for your claim. The form must clearly explain the facts and reasons why the insurer believes your injury or illness did not result from your work.

- If you disagree with the denial, you should talk with the insurance claims adjuster who is handling your claim. Your employer's insurance company can answer most questions about your claim.

Insurer name: Zurich North America
Fax# 1 (877) 962-2567

Phone : 1 (800) 987-3373
http://www.zurichna.com

- If you are not satisfied with the response you receive from the insurer and still disagree with the denial, you should contact the Department of Labor and Industry at one of the numbers listed below to see what to do next.

If you have other questions or need more help, call the Minnesota Department of Labor and Industry Workers' Compensation Hotline:

Twin Cities and Southern Minnesota: **(651) 284-5005 or 1-800-342-5354; TTY (651) 297-4198**
Duluth and Northern Minnesota: **(218) 733-7810 or 1-800-365-4584**

Your call will be answered by experienced workers' compensation specialists, who will provide **instant, accurate information and assistance.**

Additional workers' compensation information is available on the department's Web site at:
www.doli.state.mn.us

Your employer is required by law to give you this information. This material can be made available in different formats, such as large print, Braille or on audiotape, by calling the numbers printed above.

Updated April 2003 (format-change only).

Bay West
Customer-Focused Environmental & Industrial Solutions

Near Miss Investigation Report

Instructions: A near miss is an unplanned event that did not result in injury, illness, or damage – but had the potential to do so. Only a fortunate break in the chain of events prevented an injury, fatality or damage. In the event of a near miss, take appropriate action to correct the unsafe condition and submit this completed form as soon as practical, but no later than **48 hours** after the near miss. The Safety and Health Manager (SHM) will complete a follow-up investigation and awareness training. **Remember:** Everyone has the right and responsibility to stop work in the event of an unsafe condition. Questions regarding this form can be directed to the SHM.

Date of Report:		Time of Report:	
Person completing this form:		Signature:	
Person(s) involved:			
Witnesses:			
Project Manager:		BW Job #	J
Date of near miss:		Time of near miss:	
Nature of the near miss	☐ Unsafe Act ☐ Unsafe Condition ☐ Unsafe Equipment ☐ Near Collision		
What is the likelihood of a similar near miss happening?	☐ Almost Certain ☐ Likely ☐ Possible ☐ Unlikely ☐ Rare		
What level of consequence could such a near miss event have?	☐ Catastrophe ☐ Major ☐ Moderate ☐ Significant ☐ Negligible		
Where did the near miss occur?			
What happened? – Describe the near miss:			
Why did the near miss occur? (In your opinion):			
Was work stopped as a result of the near miss?	☐ Yes ☐ No		
Have unsafe conditions been corrected?	☐ Yes ☐ No		
If yes, what has been done? What further actions can be made?			
If no, what needs to be done?			

Schematic / Notes: Provide visual aids or additional notes for the persons involved in the near miss investigation. Include photographs, sketches, or graphics to document the location and how the near miss occurred.

Appendix 6

Prevention of Alcohol and Drug Abuse Plan

5.0 DRUG AND ALCOHOL USE AND TESTING PROGRAM

5.1 Introduction

Bay West recognizes that alcohol and drug abuse adversely affects employees' safety and health, job performance, their employment opportunities, and the quality of their work. While Bay West has no intention of intruding into the private lives of its employees, it strongly believes that a drug-free workplace is in the best interest of both the employee and Bay West alike and is committed to complying with all applicable federal, state, and local rules and regulations concerning the prevention of drug and alcohol use in the workplace.

5.1.1 Purpose

The purpose of this anti-drug and alcohol misuse plan is to provide employees with company policy regarding the use of drugs and alcohol while at work. Therefore, in accordance with these policies, Bay West has developed the following Drug and Alcohol Use and Testing Program. The intent of this Policy is to comply with DOT requirements for CMV drivers (49 CFR Part 40 *FMCSA Procedures for Transportation Workplace Drug and Alcohol Testing Programs),* emergency response work on pipeline spills of hazardous materials (49 CFR Part 199 *PHMSA Drug and Alcohol Testing*), and safety-sensitive positions per Minnesota's Drug and Alcohol Testing in the Workplace Act (DATWA), Minn. Stat. 181.950 et. seq.

5.1.2 Scope

This policy applies to all covered employees including applicants for employment, and to all operational, administrative, contract, or temporary employees. The policy is applicable at Bay West facilities or whenever employees are performing company business. This policy applies whenever anyone is representing or conducting business for the organization. Therefore, this policy applies during all working hours, whenever conducting business or representing the organization, while on-call, or while on paid standby.

5.1.3 Disclaimer

Employment at Bay West is at-will. This policy is not a unilateral employment contract and should not be interpreted as creating a unilateral employment contract.

5.1.4 Non-Discrimination

The policy on work-related substance abuse is non-discriminatory in intent and application. However, in accordance with Minnesota Statutes, disability does not include any condition resulting from alcohol or other drug abuse that prevents a person from performing essential functions of the job or creates a direct threat to property or the health or safety of individuals.

5.1.5 General Prohibitions

- No employee shall report to work under the influence of alcohol, marijuana, controlled substances, or other drugs that affect his/her alertness, coordination, reaction, response, judgment, decision-making, health, or safety.
- No employee shall operate, use, or drive any equipment, machinery, or vehicle of Bay West while under the influence of alcohol, marijuana, controlled substances, or other mood-altering drugs. Every employee is under an affirmative duty to immediately notify his/her supervisor if he/she is not in an appropriate mental or physical condition to operate, use, or drive company owned equipment.
- No employee shall unlawfully manufacture, distribute, dispense, possess, transfer, or use

marijuana, controlled substances, or other drugs in the workplace or wherever Bay West's work is being performed.

- Engaging in off-duty sale, purchase, transfer, use, or possession of illegal drugs or controlled substances may have a negative effect on an employee's ability to perform his/her work for Bay West. In such circumstances, the employee is subject to disciplinary action.
- When an employee is taking medically authorized drugs or other substances that may alter job performance, the employee is under an affirmative duty to notify the appropriate supervisor if he/she is temporarily unable to perform the job duties of his/her position.
- Bay West will notify the appropriate law enforcement agency when it has reasonable suspicion to believe that an employee may have illegal drugs in his/her possession at work or on Bay West premises. When appropriate, Bay West will also notify licensing boards.
- Employees are prohibited from consuming alcoholic beverages during lunch periods, dinner periods, or breaks when the employee intends on returning immediately thereafter to perform work on behalf of Bay West.

5.2 Definitions

Adversely Affected Work Performance and Under the Influence refer to situations when an employee is perceptively impaired, has impaired alertness, coordination, reactions, responses, or efforts; if the employee's condition threatens the safety of him/herself or others; or if the employee's condition or behavior presents the appearance of unprofessional or irresponsible conduct detrimental to the public's perception of Bay West as an employer as determined by the supervisor or manager or other observing the employee.

Alcohol is an intoxicating agent in beverage alcohol, ethyl alcohol, or other low molecular weight alcohols including methyl and isopropyl alcohol.

Alcohol use refers to the consumption of any beverage, mixture, or preparation, including any medication containing alcohol.

Commerce is defined as any trade, traffic, or transportation within the jurisdiction of the U.S. (a) between a place in a State and a place outside of such State, including a place outside of the United States, and (b) trade, traffic, and transportation in the United States which affects any trade, traffic, and transportation described above.

Commercial motor vehicle (CMV) is a motor vehicle used in commerce to transport passengers or property if the motor vehicle meets any of the following criteria:

a. Has a Gross Combination Weight Rating (GCWR) of 26,001 or more pounds inclusive of a towed unit with a Gross Vehicle Weight Rating (GVWR) of more than 10,000 pounds; or
b. Has a GVWR of 26,001 or more pounds; or
c. Is designed to transport 16 or more passengers, including the driver; or
d. Is of any size and used in transportation of materials found to be hazardous for the purposes of the Hazardous Materials Transportation Act (HMTA) and which require the motor vehicle to be placarded under the Hazardous Materials Regulations (HMR) (49 CFR PART 172, subpart F).

Driver refers to any person who operates a CMV. This includes, but is not limited to full time, regularly-employed drivers and independent owner-operator contractors who are either directly employed by or under lease to an employer or who operate a CMV vehicle at the direction of or with the consent of an employer. For the purpose of pre-employment/pre-duty testing only, the term "driver" includes a person applying to an employer to drive a CMV.

Controlled Substances are substances whose distribution is controlled by regulation or statute,

including but not limited to narcotics, depressants, stimulants, hallucinogens, and cannabis.

Mood-Altering or Altered refers to changed behavior that may limit an employee's ability to safely and efficiently perform his/her job duties or poses a threat to the safety of the employee or others.

Incidents

1. **Liquid Natural Gas (LNG) Incident**
   - An event that involves a release of gas from a pipeline or if liquefied natural gas, or gas from an LNG facility;
   - A death or personal injury necessitating in-patient hospitalization;
   - Estimated property damage, including cost of gas lost, of the operator or others or both, of $50,000 or more;
   - An event that results in an emergency shutdown of an LNG facility; or
   - An event that is significant in the judgment of the operator.

2. **Hazardous Liquid Pipeline Incident**

   Any failure in a pipeline system in which there is a release of the hazardous liquid or carbon dioxide transported resulting in any of the following:
   - Explosion or fire not intentionally set by the contractor;
   - Loss of 50 or more barrels of liquid, or carbon dioxide; or
   - Escape to the atmosphere of more than five barrels a day of highly volatile liquids.

3. **Death of Any Person**
4. **Bodily harm to any person resulting in one of the following:**
   - Loss of consciousness;
   - Necessity to carry the person from the scene;
   - Necessity for medical treatment; or
   - Disability that prevents the discharge of normal duties or the pursuit of normal activities beyond the day of the incident.

5. **Estimated property damage to the property of the contractor or others, or both exceeding $5,000.00 when working on a pipeline project.**

Safety-sensitive function Refers to any of those on-duty functions set forth in §395.2 On-Duty time, 49 CFR part 172, subpart F.

Safety-sensitive position means a job, including any supervisory or management position, in which an impairment caused by drug or alcohol usage would threaten the health or safety of any person.

Work-Related Alcohol and Other Drug Abuse refers to the use of mood-altering drugs, including all forms of alcohol, narcotics, depressants, stimulants, hallucinogens, marijuana, or the use of prescription drugs (R_x) when resulting behavior or appearance adversely affects work performance.

5.3 Affected Employees, Designated Bay West Representatives

All Bay West employees and applicants will be notified of the provisions of the Bay West Drug and Alcohol Use and Testing Program by receiving a copy of this Plan. In addition, copies of the Plan are available from the SHM. Each employee receiving a copy of the Plan will sign the Agreement and Consent to Drug and/or Alcohol Testing form (**Appendix 5**).

The job titles of Bay West supervisory personnel that receive "Reasonable Suspicion" training include:

- Vice President of Operations

- Team Leaders
- PMs
- SHM
- Human Resources Manager

The job titles of Bay West personnel responsible for recordkeeping include:

- SHM
- Human Resources Manager
- Human Resources Assistant

5.4 Confidentiality

Bay West utilizes seven methods to ensure employee confidentiality.

5.4.1 Seven Methods for Insuring Confidentiality of Records

1. A Medical Review Officer (MRO) receives the laboratory reports and communicates the information to the SHM via mail marked "CONFIDENTIAL," and a secure e-mail. The information is printed and filed in a locked file. Electronic correspondence that is not password protected is deleted.
2. Mail marked "CONFIDENTIAL" is not opened by the receptionist.
3. Mail marked "CONFIDENTIAL" addressed to the SHM is opened by the SHM, and the results are filed in a locked employee drug testing file.
4. An employee who asks to review his/her drug testing records is allowed to examine the records in the presence of the SHM or Human Resources Manager. An employee may make a copy of their drug testing record.
5. The employee drug testing file is kept locked unless records are added, records are reviewed for reporting purposes, or for employee examination. Password protected drug testing results are only accessible by the Human Resources Manager, Human Resources Assistant, and the SHM.
6. Records are not removed from the room where the locked file is located.
7. Removed records and unlocked files are not to be left unattended. Removed records are replaced or covered when other personnel enter the area.

5.4.2 Ensuring Confidentiality of Positive Test Results

Positive test results (as determined by the MRO) will be communicated to the SHM and the Human Resources Manager. The SHM will inform the "employer" of the positive test results. The employer includes the employee's immediate supervisor and Bay West management team members having a specific need to know. Each person receiving information regarding an employee's positive drug test will be expected to maintain strict confidentiality of the information.

5.5 Controlled Substances

The following five DOT prohibited drugs for which employees will be tested include:

- Marijuana
- Cocaine
- Amphetamines
- Phencyclidine
- Opiates

5.6 Tests Conducted

Employees who are required to submit to testing under this program must appear at the designated collection site at the time of their appointment as directed by the Human Resources Assistant, Human Resources Manager, or SHM. In order to carry out the Company's commitment to an alcohol and drug-free workplace, Bay West reserves the right to require that applicants and employees submit to testing in accordance with the provisions of the applicable state in which the employee is employed. This policy represents the notice required under Minnesota Statute and will be provided to all applicants and employees who are requested to undergo testing. Testing for out of area employees will be arranged through a local occupational health center.

5.6.1 Who May be Subject to Testing

5.6.1.1 Job Applicants

Bay West will require all job applicants who have received conditional offers of employment and will be placed into a random testing pool to be tested. If the offer of conditional employment is subsequently withdrawn, Bay West will notify the applicant of the reason for the withdrawal.

5.6.1.2 Routine Physical Examination Testing

Bay West may require employees to undergo testing, not more than once a year, as part of a routine physical examination. Employees to be tested will be notified at least two weeks in writing in advance of the examination.

5.6.1.3 Random Testing

Employees in the FMCSA, PHMSA, and safety-sensitive position pools are randomly selected for testing through Trust in Us Drug, Alcohol and DNA Testing. Employees are instructed to proceed to the testing facility within one hour of notification. If the employee is out of the office at the time of notification, he/she will be notified upon arrangement of the closest qualifying clinic.

Random selection is conducted quarterly throughout the year. The selection mechanism, in each pool, will be such that every employee has an equal probability of being selected. The number of employees selected for random testing in a year will be equal to: 25% of the eligible employee pool for drugs under the PHMSA program, 25% of the eligible employee pool for drugs under the safety-sensitive position pool, and 50% of the eligible employee pool for drugs and 10% for alcohol under the FMCSA program.

Random Testing Pools

Bay West employees subject to random testing will be placed into one of following three pools.

FMCSA

- All Bay West employees that hold a valid CDL

PHMSA

- All Bay West employees, that are not in the FMCSA pool, with the following designations:
  - Emergency Response Manager
  - Emergency Response Technician
  - Emergency Response On-call, including part time on-call, leads and back-ups (i.e. on the on-call schedule)

Safety-sensitive Position

- If not already subject to a DOT testing pool (FMCSA or PHMSA), Bay West employees with the following titles are considered to be in a safety sensitive position and will be placed in a Non-DOT Safety-sensitive position pool.
  - Unexploded Ordnance (UXO)
    - Senior UXO Supervisor (SUXOS)
    - UXO Quality Control Specialist (UXOQCS)
    - UXO Safety Officer (UXOSO)
    - UXO Technician (I, II, and III)
  - Automotive Technician

5.6.1.4 Reasonable Suspicion Testing

Bay West may require an employee to be tested when Bay West reasonably suspects that the employee is:

- Under the influence of drugs or alcohol;
- Has violated the Company's written work rules prohibiting drug and alcohol use;
- Has sustained or caused another employee to sustain personal injury; or
- Has caused a work-related incident or was operating or helping to operate machinery, equipment, or vehicles involved in a work-related incident.

5.6.1.5 Post Incident

Employees subject to post-incident testing must remain available for such testing. If not, the employee may be deemed to have refused testing. If a post-incident test for alcohol is not administered within two (2) hours of the incident, Bay West will prepare and maintain a record stating why the test was not administered. If a post-incident alcohol test cannot be administered within eight (8) hours, Bay West will stop any attempts to administer the test, and will prepare and maintain a record stating why the test was not administered. An employee will be tested for controlled substances no later than thirty-two (32) hours of an above-referenced incident. If a post-incident test for drugs is not administered within thirty-two (32) hours of the incident, Bay West will prepare and maintain a record stating why the test was not promptly administered. If the employee is too seriously injured to provide a urine sample, he or she must authorize release of hospital records.

5.6.1.6 Return to Duty

Employees who return to duty after rehabilitation shall be subject to a reasonable program of follow up drug testing without prior notice during 60 months after his/her return to duty.

5.7 Conducting the Testing

5.7.1 Consent

All persons to be tested will be required to complete and sign the employee consent form **Appendix 5**. Each signed form must also be witnessed by a designated person within the Company.

5.7.2 Refusal to Participate

An employee or job applicant has the right to refuse testing. However, a refusal of testing will be treated as a failure to comply with Bay West policy and may result in withdrawal of a job offer or disciplinary action up to and including termination of employment.

5.7.3 The Laboratory

The laboratory selected to perform testing must be certified by the National Institute on Drug Abuse (NIDA), the College of American Pathologists (CAP), or the New York State Department of Health.

5.7.4 Test Results

5.7.4.1 Negative Test Results (passed tests)

Individuals who test negative on an initial drug or alcohol test will be given written notice of the test result within three (3) days after Bay West is notified of the result. Likewise, individuals who test negative on a confirmatory test taken after a positive initial test will be given written notice of the test result within three (3) days after Bay West is notified of the result.

5.7.4.2 Positive Test Results (failed tests)

A confirmatory test will automatically be performed on all samples that result in a positive test result on an initial test. Individuals who test positive on the confirmatory test will be notified in writing of the test result and of the right to explain the result, including any over-the-counter or R_x they have taken, within three (3) days after Bay West has been notified of the result.

Individuals who wish to provide explanatory information regarding their positive confirmatory test result must do so in writing within (3) three working days after receiving notice of the positive test result.

Individuals who wish to have a *retest* of their confirmatory test must notify Bay West in writing of their intention to have a retest within five (5) working days after being notified of the confirmatory test result. Persons requesting a confirmatory retest are responsible for the cost of the retest.

5.7.4.3 Right to Test Result

An employee or job applicant has the right to request and receive from Bay West a copy of the test result report on any drug or alcohol test.

5.7.5 Costs

All costs related to alcohol and drug testing will be paid by Bay West, with the exception of confirmatory retests, which must be paid for by the employee or job applicant requesting the retest.

5.8 Disciplinary Action in Response to a Positive Test Result

5.8.1 Interim Disciplinary Action

Bay West reserves the right to transfer an employee with a positive test to another position at the same rate of pay or to temporarily suspend the employee pending the outcome of the confirmatory

test (and, if requested, the confirmatory re-test) if Bay West believes that it is reasonably necessary to do so. An employee who is suspended without pay will be reinstated with back pay if the confirmatory test or re-test is negative. In the case of job applicants, a positive initial test result must be verified by a confirmatory test before a conditional offer of employment will be withdrawn.

5.8.2 First Failed Test – Discharge/Withdrawal of Job Offer

Bay West will not discharge an employee if the employee tests positive on a confirmatory test and the positive confirmatory test was the first such result. Bay West may, however, discharge an employee for whom a positive confirmatory test is the first such result where:

1. The employee is given an opportunity to participate in, at the employee's expense or pursuant to coverage under an employee's benefit plan, a drug or alcohol counseling or rehabilitation program; and
2. The employee has either refused to participate in the counseling or rehabilitation program or has failed to successfully complete it.

The type of counseling or rehabilitation program in which an employee participates will be determined by Bay West after consultation with a certified chemical use counselor or physician trained in the diagnosis and treatment of chemical dependency. Bay West reserves the right to withdraw a conditional offer of employment from a job applicant who tests positive on a confirmatory alcohol and/or drug test.

5.8.3 First Failed Test – Discipline

Bay West reserves the right to take any other disciplinary action short of discharge it deems warranted in the event that an employee tests positive on his or her first confirmatory test.

5.8.4 Second Failed Test

Bay West reserves the right to discharge an employee who tests positive on a confirmatory test and who has previously had a positive confirmatory test result. This action may be taken without first referring the employee to a chemical dependency counseling or rehabilitation program.

5.9 Privacy of Test Results

Test results and other information acquired as a result of the testing program are private and confidential information and will not be disclosed by Bay West or the testing laboratory to another employee or to third party individuals, government agencies, or private organizations without written consent of the employee or applicant being tested.

Evidence of a positive test result on a confirmatory test, however, may be used in an arbitration proceeding pursuant to a collective bargaining agreement, an administrative hearing, or a judicial proceeding, provided the information is relevant to the hearing or proceeding. Such evidence may also be disclosed to any federal agency or other unit of the United States government as required under federal law, regulation, or order. Evidence of a positive test result on a confirmatory test may also be disclosed to a substance abuse treatment facility for the purpose of evaluation or treatment.

Bay West will provide an employee with access to information in the employee's file relating to positive test result reports and other information acquired in the testing process as well as conclusions drawn from or actions taken based upon such information.

5.10 Testing Laboratory

The drug-testing laboratory is:

Clinical Reference Laboratory

8433 Quivira Road
Lenexa, KS 66215
913-492-3652

The testing laboratory is required to retain samples that yield confirmed positive test results for one year in frozen, secured storage.

5.11 Medical Review Officer

The MRO reviews all results (negative and positive) before they are reported to the company. The MRO is Doctor Stephen Kratch, MD. Dr. Kratch is a licensed physician with knowledge of drug abuse disorders. His address is:

eScreen, Inc
7500 W. 110th Street Suite 500
Post Office Box 25902
Overland Park, KS 66225-5902
866-282-2281 fax: 913-469-4029

The MRO reviews and interprets positive test results as described below to determine if there is an alternative medical explanation for a confirmed positive test result.

1. The MRO conducts a medical interview;
2. The MRO reviews the medical history and any relevant biomedical factors;
3. The MRO reviews all medical records to determine if a confirmed positive test resulted from legally prescribed medication;
4. If necessary, the MRO authorizes a reanalysis of the original specimen to reaffirm the accuracy of the test results;
5. The MRO verifies that the laboratory report and assessment are correct;
6. The MRO performs the following duties in situations where employees who have tested positive may eventually return to duty:
   - The MRO determines whether, and when, the employee may be returned to duty.
   - The MRO determines the schedule for testing after an employee has been slated for return to duty.
   - The MRO ensures that the employee has been drug tested in accordance with DOT procedures before the employee is returned to duty.
7. The MRO uses the following rules to make determinations:
   - The MRO takes no further action if there is a legitimate reason for a positive drug test and reports the test as negative.
   - If there is no legitimate reason for a confirmed positive drug test, the MRO refers the individual tested to the SHM or Human Resources Manager for action in accordance with this plan.
   - A specimen deemed scientifically insufficient by the MRO will be reported by the MRO to Bay West as a negative test result. Before declaring the scientifically insufficient specimen negative, the MRO must consider the following:
     a. Reanalysis of the original sample performed by the same laboratory, or
     b. Reanalysis of the original sample to an alternate laboratory which is certified in

accordance with the DHHS Guidelines, or

c. Consultation from the drug testing laboratory concerning the drug test results.

5.12 Consequences of a Failed Test

"Failing a drug test" means the MRO has determined there is no legitimate medical explanation for the confirmed positive test other than unauthorized use of a prohibited drug or alcohol.

If an applicant for a covered position fails a test, his/her conditional offer of employment may be withdrawn.

If an employee fails a test he/she will be unqualified to participate in covered or safety sensitive activities (maintenance or emergency response operations at LNG/pipeline release sites, DOT commercial driving, etc.).

5.13 Consequences of Refusal to Take a Test

An employee or an applicant has the right to refuse to submit to drug and/or alcohol testing. If the employee or applicant does not present him/herself at the appointed place and time for drug and/or alcohol testing, this will be construed as refusal. If an employee refuses to submit to drug and/or alcohol testing, he/she will be unqualified to participate in covered or safety sensitive activities, and he/she will be subject to disciplinary action up to and including immediate termination. If an applicant refuses to submit to drug and/or alcohol testing, his/her offer of employment may be withdrawn.

Use of an employee in a covered or safety sensitive position is acceptable under the following conditions:

1. The employee has passed a test following a positive test;
2. The employee has been recommended by the MRO for Return to Duty; and
3. The employee has not failed a test after being returned to duty following a positive test.

Within five (5) working days of receipt of final (positive) test results from the MRO, the employee or applicant may notify Bay West in writing of his/her intention to obtain a confirmatory test at his/her expense. The original sample may be analyzed by the same laboratory or transferred under NIDA-approved chain-of-custody procedures to another DHHS- certified laboratory for analysis. If the confirmatory test does not confirm the original positive test result, no adverse personnel action based on the original confirmatory test may be taken against the employee or applicant. If the retest is negative, the employee or applicant will be reimbursed for the retest cost.

Because it is possible that some analytes may deteriorate during storage, the result of a retest is to be reported as confirmation of the original test result even if the detected level of the drug is (a) below the DOT established limits and (b) equal to or greater than the sensitivity of the test.

5.14 Rehabilitation

This section applies only to Bay West employees (not applicants):

In the event of a confirmed positive test result, the Human Resources Manager will meet with the affected employee to discuss evaluation and rehabilitation. An employee for whom a positive test result was the first such result will be offered an opportunity to participate, at the employee's own expense, either a drug or alcohol counseling or rehabilitation program, whichever is more appropriate after consultation with a certified chemical use counselor or a physician trained in the diagnosis and treatment of chemical dependency. The employee will be responsible for covering all costs associated with the initial consultation and evaluation of the employee with a certified drug and/ or alcohol counselor. The employee has the opportunity to participate, at his/her own expense, in a drug

and/or alcohol counseling or rehabilitation program, as determined by Bay West through consultation with trained and certified counselors or physicians.

Employees shall be given access to information in their personnel file relating to positive test result reports and other information acquired in the testing process and conclusions drawn from, and actions taken, based on the reports and other acquired information.

No other appeal procedures are available to employees.

5.15 Discipline and Discharge

A covered employee who tests positive twice for alcohol or any of the five classes of drugs, regardless of the time span between positive tests, may be terminated at the discretion of Bay West management.

An employee may be terminated if he/she refuses to participate in the counseling or rehabilitation program or has failed to successfully complete the program, as evidenced by a withdrawal from the program before its completion or by a positive test result on a confirmatory test any time after completion of the program.

5.16 Employee Assistance Program

Informational material is displayed and distributed to employees regarding how to access assistance and the Bay West Drug and Alcohol Use and Testing Program.

Counseling for drug or alcohol dependency is available through the Bay West medical insurance provider on either an inpatient or outpatient basis depending what is recommended by the counseling professional.

As part of the Employee Assistance Program (EAP), Bay West provides initial training to covered employees on the hazards of substance abuse and initial training to supervisors (who are authorized to initiate "reasonable cause" testing) on the health effects of drug use, treatment options, signs and symptoms of abuse, and intervention strategies in the work place.

5.17 Recordkeeping

The records identified below are kept for the specified periods and these records verify that the policies of the Bay West Drug and Alcohol Use and Testing Program are being followed.

a. Records that demonstrate that the DOT collection process conforms to 49 CFR Part 40.25 (Specimen Collection Procedures) are kept for 3 years. These records consist of the employer copy of the chain-of-custody form.
b. Records of employee test results that show employees failed a test, and the type of test failed, and records that demonstrate rehabilitation, if any, are kept for at least 5 years, and include the following information:
    1. The functions performed by the employee who failed the test.
    2. The prohibited drugs and/or alcohol that were detected in the specimen of the employee who failed the test.
    3. The disposition of the employee who failed the test.
    4. The age of each employee who failed a test.
c. Records of employee test results that show employees passed tests shall be kept for at least one year.
d. A record of the number of employees tested by type of test (e.g., random, pre-placement) are kept for at least 5 years.

e. Records confirming that supervisors and employees have been trained as required by 49 CFR Part 40 are kept for least 3 years.

f. Release of an individual's test results is prohibited except as identified below:
   1. Upon written request of the individual
   2. Upon request by the DOT, a state agency, or a federal agency pursuant to:
      - An incident investigation
      - Statistical evaluations (with no individual names required)

5.18 Specimen Collection

5.18.1 Regulations

Urine specimens collected under DOT regulations may ONLY be used to test for controlled substances designated or approved for testing by the DOT and shall not be used to conduct any other analysis or test unless otherwise specifically authorized by the DOT.

Drug testing custody and control form (CCF) by the Office of Management and Budget (OMB) No. 0930-0158 or equivalent will be utilized for DOT testing only. Non-DOT testing will conducted using the same procedure as DOT, but recorded on a non-DOT CCF.

5.18.2 Provisions for Collection

Bay West has designated and approved collection sites which will have personnel, materials, equipment, facilities, and supervision necessary to provide for collection, security, temporary storage, and shipping of urine specimens to the laboratory.

A specimen will be collected at the approved collection site which has an enclosure within which private urination can occur, a toilet for completion of urination (unless a single-use container is used with sufficient capacity to contain the void), and a suitable clean surface for writing. The site must also have a source of water for washing hands, which if practical, should be external to the enclosure where urination occurs.

The collection site is not dedicated solely to specimen collection.

If the sample is being collected from an employee in need of medical attention (e.g., as part of a post-incident test given in an emergency medical facility), necessary medical attention shall not be delayed in order to collect the specimen.

Bay West specimen donors are provided written standard instructions setting forth their responsibilities during specimen collection.

Collection site personnel provide the specimen donor with a clean single-use specimen bottle that is securely wrapped.

Collection site personnel provide a tamper-proof sealing system designed in a manner to ensure against undetected opening.

A shipping container is provided in which the specimen and associated paperwork is transferred after being sealed and initialed to prevent undetected tampering.

5.18.3 Specimen Security

If continuous physical security is impractical at the collection site from the time the specimen is presented until the sealed mailer is transferred for shipment:

- Specimen shall remain under the direct control of a collection site person from submission to its being sealed in the mailer.

- Mailer shall be immediately mailed, maintained in secure storage, or remain until mailed under the personal control of a collection site person.

5.18.4 Suspicion of Intent to Alter Specimen

Collection site personnel shall allow individual privacy unless there is reason to believe that an individual has intent to alter or substitute a specimen. The following circumstances are the exclusive grounds constituting a reason to believe that an individual may alter or substitute a specimen:

- Individual presents a specimen that falls outside the normal temperature range of 32.5 to 37.7 degrees Centigrade (°C) or 95.5 to 99.8 degrees Fahrenheit (°F), and

  The employee declines to provide a measurement of oral body temperature; or
  Oral body temperature varied by more than 1°C/1.8°F from the temperature of the specimen.
- The last urine specimen provided by the individual was determined by the laboratory to have a specific gravity of less than 1.003 and a creatinine concentration below 0.2 grams per liter (g/L). The collection site person observes an attempt to substitute or adulterate the sample.
- Employee has previously tested positive on a drug test and is having a follow-up RETURN TO DUTY test.

If the employee refuses to cooperate with the collection process, the collection site person shall inform the employer representative and shall document the non-cooperation on the drug testing CCF.

5.18.5 Specimen Handling Procedure

5.18.5.1 Before Specimen Collection

1. Bluing agents in toilet tank and all water sources will be secured.
2. Contact the proper authority (SHM) if the individual fails to arrive at the assigned time.
3. Individual should be positively identified with photo ID.
4. The donor shall remove any unnecessary outer garments. Purses or briefcases shall remain with outer garments.
5. Donor shall wash and dry his/her hands.

5.18.5.2 During Specimen Collection

6. The individual shall remain in the presence of the collection site person and shall not have access to water or any other materials which could be used to adulterate the specimen.
7. The donor shall have individual privacy.
8. Any unusual behavior should be noted on the CCF.
9. Provisions have been made if the donor is unable to provide at least 60 milliliters (mL) of urine, especially in reference to Post-Incident and Reasonable Cause testing (i.e., donor drinks reasonable quantities of fluid for up to 8 hours).

5.18.5.3 After Specimen Collection

10. Donor shall be allowed to wash his/her hands after the specimen is collected.
11. Temperature of the specimen must be taken within 4 minutes of specimen collection.
12. If specimen temperature is outside the normal range (32.5 to 37.7 °C or 90.5 to 99.8 °F), oral temperature may be taken from the donor if the donor agrees.

13. Inspect specimen for any contaminants and note findings on CCF.
14. Specimens suspected of being adulterated must be forwarded to the laboratory for testing.
15. If there is a reason to believe the specimen is adulterated or substituted, the donor must, as soon as possible, provide a second specimen under direct observation of a same-gender collection person.
16. The specimen must be in view at all times before being sealed and labeled.
17. Both the specimen donor and the collector are present during the collection process.

5.18.5.4 Documentation of Specimen

18. Identification label must be placed on the specimen container.
19. Donor must initial the identification label on the specimen bottle.
20. The collection site person must enter the identifying information of the specimen on the CCF and shall sign the form for certification that Federal requirements have been met.
21. The donor reads and signs the CCF to verify that the specimen identified is, in fact, the specimen that the donor provided.
22. The CCF is completed after receipt of the specimen from the donor.

5.18.5.5 Security and Shipping of Specimen

23. The specimen and the CCF are prepared for shipment or are safeguarded during temporary storage.
24. The collector must have control of the CCF throughout the performance of the collection procedure. If the collector leaves the workstation, the CCF and the specimen must go with him/her or they must be secured.
25. The collector shall not leave the workstation between specimen presentation and securement of sample. If collector leaves, the collection is nullified and the collection starts over if the employer elects to do so.
26. Collection site personnel shall arrange to ship the collected specimens to the drug testing laboratory by placing the specimens in shipping containers designed to minimize the possibility of damage during shipment, and those containers shall be securely sealed to eliminate the possibility of undetected tampering. On the tape sealing the container, the collection site person shall sign and enter the date specimens were sealed in the containers for shipment. The collection site person shall ensure that the chain-of-custody documentation is attached to each container sealed for shipment to the drug testing laboratory.

5.19 Laboratory Report

The laboratory report submitted to the MRO identifies the following items:

- Drugs/metabolites for which the sample was tested;
- Test result: negative or positive;
- Specimen number assigned; and
- Drug testing laboratory specimen identification number (accession number).

The laboratory shall report as negative all specimens which are negative on the initial test or negative on the confirmatory test as negative and only specimens confirmed positive shall be reported positive

for a specific drug.

In the case of a positive report for drug use, the CCF is signed by the individual responsible for day-to-day management of the drug testing laboratory or the individual responsible for attesting to the validity of the test reports.

5.19.1 Quarterly Statistical Summary

Bay West receives a Quarterly Statistical Summary containing the following information:

Initial testing

- Number of specimens received;
- Number of specimens reported out;
- Number of specimens screened positive for:
  - Marijuana metabolites
  - Cocaine metabolites
  - Opiate metabolites
  - Phencyclidine
  - Amphetamines

Confirmatory testing

- Number of specimens received for confirmation;
- Number of specimens confirmed positive for:
  - Marijuana metabolite
  - Cocaine metabolite
  - Morphine, Codeine
  - Phencyclidine
  - Amphetamine
  - Methamphetamine

NOTE: The laboratory will not send the Statistical Report if the report includes data from which it is reasonably likely that information about an individual's tests can be readily inferred.

Bay West submits three blind performance test specimens for each 100 drug tests completed up to a maximum of 100 blind performance test specimens submitted per quarter.

5.20 Reporting Positive and Negative Test Results

After making all reasonable efforts and documenting them, if the MRO is unable to reach the individual directly, the MRO contacts the SHM or Human Resources Manager who will then direct the individual, in strict confidence, to contact the MRO as soon as possible.

5.20.1 Positive Results

The MRO can verify a test as positive without having communicated directly with the employee/job applicant about the test in the following circumstances:

- The employee/job applicant expressly declines the opportunity to discuss the test.
- The SHM or Human Resources Manager has successfully made and documented a contact with the employee/job applicant and instructed the employee/job applicant to contact the MRO and more than 5 days have passed since the date the employee/job applicant was successfully contacted.
- Other circumstances provided for in DOT agency drug testing regulations.
- If an employee/job applicant contacts the MRO after the above-mentioned 5-day time period and presents an acceptable explanation as to why previous contact with the MRO was not made, the

MRO will reopen the case and allow the individual to present information concerning legal drug use resulting in a confirmed positive test and, if the MRO concludes that there is legal drug use, the MRO will declare the test to be negative.

- If any questions arise as to the accuracy or validity of a positive test result, the MRO is the only person authorized to order a reanalysis of the original sample.
- The MRO will authorize a reanalysis of the original sample if requested to do so by the employee/job applicants having received actual notice of the positive test.

5.20.2 Negative Results

Before declaring the test specimen negative, the MRO must consider the following:

- Request a reanalysis of the original sample performed by the same laboratory;
- Request a reanalysis of the original sample to an alternate laboratory which is certified in accordance with the DHHS Guidelines; and
- Receive specific consultation from the drug testing laboratory concerning the drug testing results.

5.20.3 Confidentiality

The MRO does not disclose to Bay West, a DOT agency or other Federal safety agency, or a physician responsible for determining qualification of the employee/job applicant under an applicable DOT agency regulation, any medical information provided by the individual to the MRO as part of the testing verification process except as provided below:

- An applicable DOT regulation permits or requires such disclosure;
- In the MRO's reasonable medical judgment, the information could result in the employee/job applicant being determined to be medically unqualified under an applicable DOT agency rule; and
- In the MRO's reasonable medical judgment, in a situation in which there is no DOT agency rule establishing physical qualification standards applicable to the employee/job applicant, the information indicates that continued performance by the employee/job applicant of his or her safety-sensitive function could pose a significant safety risk.

Before obtaining medical information from the employee/job applicant as part of the verification process, the MRO informs the employee/job applicant that information may be disclosed to third parties as provided above, and the MRO must identify any third party to whom information may be disclosed.

The testing laboratory maintains test records in confidence as provided in DOT agency regulations.

The contract with the testing laboratory provides that the laboratory shall disclose information related to a positive drug test of an individual to the individual, the employer, or the decision-maker in a lawsuit, grievance, or other proceeding initiated by or on behalf of the individual.

Upon written request, the employee/job applicant has access to any records relating to his or her drug test and any records relating to the results of any relevant certification, or revocation-of-certification proceedings.

Attachment A

Site Safety and Health Plan

Final
SITE SAFETY AND HEALTH PLAN
FOR
CS-C503

PERFORMANCE-BASED RESTORATION
JOINT BASE ANDREWS NAVAL AIR FACILITY WASHINGTON
CAMP SPRINGS, MARYLAND

Contract W9128F-10-D-0025 DO #0002
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VERSION: 00

Prepared for:

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Acronyms and Abbreviations

ACGIH..... American Conference of Governmental Industrial Hygienists
AHA..... Activity Hazard Analysis
AMEC..... AMEC Environment & Infrastructure
APP..... Accident Prevention Plan
ASP..... Associate Safety Professional
Bay West..... Bay West, Inc.
CIH..... Certified Industrial Hygienist
CO..... Carbon Monoxide
CRZ..... Contamination Reduction Zone
DO..... Delivery Order
DPT..... Direct Push Technology
EPA..... Environmental Protection Agency
EIT ..... Engineer-in-Training
ERP..... Emergency Response Plan
EZ ..... Exclusion Zone
ft..... feet or foot
HTRW ..... Hazardous, Toxic and Radioactive Waste
JBA ..... Joint Base Andrews Naval Air Facility Washington
mg/m^3..... milligrams per cubic meter
mph..... miles per hour
MSDS ..... Material Safety Data Sheet
NE..... Not Expressed
OSHA ..... Occupational Safety and Health Administration
PCB ..... Polychlorinated biphenyl
PE..... Professional Engineer
PEL..... Permissible Exposure Limit
PG ..... Professional Geologist
PM ..... Project Manager
POC..... Point of Contact
PPE ..... Personal Protective Equipment
ppm..... parts per million
PVC ..... Polyvinyl Chloride
SHM..... Safety and Health Manager
SOO..... Statement of Objectives
SSHO ..... Site Safety and Health Officer
SSHP..... Site Safety and Health Plan
SZ..... Support Zone
TLV..... Threshold Limit Value
TPH ..... Total Petroleum Hydrocarbons
TWA..... Time-weighted Average
USACE ..... United States Army Corps of Engineers
USAF ..... United States Air Force
Weston ..... Weston Solutions, Inc.

1.0 SIGNATURE PAGE

The following Bay West, Inc. (Bay West) project personnel, with teaming partners AMEC Environment & Infrastructure (AMEC) and Weston Solutions, Inc. (Weston) (collectively referred to as "the team"), have reviewed and have agreed to implement and comply with the requirements of the Accident Prevention Plan (APP) and this Site Safety and Health Plan (SSHP), for the duration of site activities.

Prepared by: _____ _____

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Reviewed by: _____ _____

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Approved by: _____ _____

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Vice President, Federal Programs
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2.0 SITE SAFETY AND HEALTH PLAN

2.1 Site Description and Contamination Characterization

2.1.1 Site Description

CS-C503, as designated in the Environmental Restoration Program at Joint Base Andrews (JBA), is a 50-foot-by-500-foot stormwater retention pond located near the intersection of Arnold Avenue and North Perimeter Road. The retention pond (typical water depth of 3 feet) was constructed between 1989 and 1995, according to available historical aerials. Both the pipe inlet and emergency overflow outlet were constructed at that time. Since the pond was constructed, there have been numerous maintenance efforts to keep the pond free from plant growth. In 2007, the pond was reconstructed and regraded to increase the capacity and to provide treatment. Polychlorinated biphenyl (PCB)-contaminated sediment was detected during the reconstruction. Currently, the source of the PCB contamination is not known; however, an electrical substation is located approximately 1,000 feet from the retention pond and is within the drainage area of the basin. In addition to the PCB contamination, total petroleum hydrocarbons (TPH) was also detected at low concentrations.

Table 2-1 Site Description

Site Location		**Approximate Size (Acres)**	
CS-C503		~0.5	
Topography		**Site Uses**	
☒ Forested	☒ Hilly	☐ Rural	☐ Mining
☐ Open Terrain	☐ Tillage	☒ Urban	☒ Military
☒ Lake, Pond	☐ River/Creeks	☐ Ag Business	☐ Residential
☒ Wetland	☐ Flat land	☐ Commercial	☒ Government
☒ Grassland	☐ Other	☐ Farming	☐ Recreational
☐ Arid		☐ Industrial	☐ Other
		☐ Ranching	

2.1.2 Project Description

The list of work phases below includes tasks planned to achieve the desired operational results for the Statement of Objectives (SOO). Under each phase of work, there are inherently hazardous activities that have been analyzed to identify controls that will protect the safety and health of all personnel working at and entering the project site.

- Mobilization/Demobilization of Personnel and Equipment; and
- Environmental Sampling.

2.2 Contamination Characterization

Potential Site Contaminants include:

Primary: PCBs.

Secondary: TPH.

Information concerning the toxicological properties of the expected contaminants, their route of exposure, and regulatory exposure standards for these materials are contained in **Table 2-2**.

Table 2-2 Potential Contaminants

Chemical	Exposure Limit	Route of Exposure	Symptoms of Exposure
Petroleum Hydrocarbons			
Benzene	0.5 ppm (2.5 ppm STEL)	Inhalation, skin absorption, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, skin, nose, or respiratory system; dizziness, headache, nausea, staggered gait, anorexia, lassitude (weakness, exhaustion), dermatitis, and bone marrow depression. Is a potential occupational carcinogen.
2-Butanone Methyl ethyl ketone MEK	200 ppm (300 ppm STEL)	Inhalation, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, skin, or nose; headache, dizziness, vomiting, and dermatitis.
Ethyl benzene	20 ppm	Inhalation, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, skin, or mucous membrane; headache, dermatitis, narcosis, and coma.
Naphthalene	10 ppm (15 ppm STEL) [2011 ACGIH Notice of Intended Change is 2 ppm]	Inhalation, skin absorption, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, headache, confusion, excitement, malaise (vague feeling of discomfort), nausea, vomiting, abdominal pain, irritation bladder, profuse sweating, jaundice, hematuria (blood in the urine), renal shutdown, dermatitis, optical neuritis, and corneal damage.
Xylenes	100 ppm (150 ppm STEL)	Inhalation, skin absorption, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, skin, nose, or throat; dizziness, excitement, drowsiness, incoordination, staggering gait, corneal vacuolization, anorexia, nausea, vomiting, abdominal pain, and dermatitis.
Total petroleum hydrocarbons	200 mg/m^3	Inhalation, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, nose, or throat; dizziness, drowsiness, headache, nausea, dry cracked skin, and chemical pneumonitis (aspiration liquid).
Polychlorinated Biphenyls (PCBs)			
Chlorodiphenyl	0.5 mg/m^3 [skin]	Inhalation, skin absorption, ingestion, skin and eye contact.	Exposure may cause irritation to eyes, chloracne, liver damage, and reproductive effects. Is a potential occupational carcinogen.

Notes:

- *Exposure Limits are the lower of either the Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) or American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit value (TLV).*
- *Exposure Limits are expressed as a time-weighted average (TWA) for a conventional 8-hour workday.*
- *mg/m^3 = milligrams per cubic meter*
- *ppm = parts per million*
- *NE = Not Expressed*
- *(Skin) = Potential significant contribution to the overall exposure by the cutaneous route, including mucous membranes and the eyes, by contact with vapors, liquids, and solids.*

Other hazardous agents which may present exposure to site workers are presented in **Table 2-3**.

Table 2-3 Potential Hazardous Agents

Chemical	Exposure Limit	Route of Exposure	Symptoms of Exposure
Silica	0.025 mg/m^3 (respirable)	Inhalation.	Lung fibrosis and silicosis.
Nuisance dusts	3 mg/m^3 (respirable)	Inhalation.	Respiratory system irritation.
Bentonite	0.025 mg/m^3 (respirable)	Inhalation.	Lung fibrosis and silicosis.

Notes:
- *Exposure Limits are the lower of either the OSHA PEL or ACGIH TLV*
- *Exposure Limits are expressed as a TWA for a conventional 8-hour workday.*
- *mg/m^3 = milligrams per cubic meter*
- *ppm = parts per million*

Refer to **Section 11.7.3**, Chemical Data in the APP for chemical group descriptions expected contaminants. Material Safety Data Sheets (MSDSs) are included as **Appendix 4**.

2.3 Activity Hazard Analysis

Appendix 1 contains an Activity Hazard Analysis (AHA) for each of the major activities anticipated at the project site. Each AHA will be reviewed and discussed by all site personnel involved with that specific activity. If an activity is planned for which there is no AHA, an analysis will be prepared and reviewed by site personnel. This review will be completed prior to initiating the site-specific activity, unless an emergency situation arises that requires an immediate response. Field activities planned for this project are listed in **Section 2.1.2**.

The AHAs are reviewed at safety briefings or daily operational meetings. Personnel will be encouraged to discuss hazards and identify ways they suspect an accident could occur and will be encouraged to suggest appropriate control measures. The meeting and topics discussed will be documented by the Site Safety and Health Officer (SSHO), using a *Safety and Health Meeting Report* form contained in the APP.

2.4 Staff Organization, Qualifications, and Responsibilities

Table 2-4 contains the project personnel, their involvement on the project, the organization these individuals represent, and contact information for these individuals.

Table 2-4 Lines of Authority

Name	Organization	Telephone	Cell Number	Email
Lucas Walsh	US Army Corps of Engineers (USACE) Project Manager (PM)	(402) 995-2750	-	lucas.v.walsh@usace.army.mil
David Connolly	US Air Force (USAF) Restoration Chief	(301) 981-1653	(703) 350-3057	david.connolly@afncr.af.mil
Lydia Plotz	USAF PM	(678) 478-4917	-	lplotz@portageinc.com
Marty Wangensteen	Program Manager	(651) 291-3475	(651) 341-3265	martyw@baywest.com
Shirley McMaster	Bay West PM	(218) 835-5852	(651) 341-3263	shirleym@baywest.com
Rob Heimbach	Bay West Site Lead	(651) 291-3476	(651) 338-9821	robh@baywest.com
Doug Hickey	Bay West Certified Industrial Hygienist (CIH)	(763) 479-3214	(612) 719-9922	dough@baywest.com
Dan Musser	Bay West Safety and Health Manager (SHM)	(651) 291-3457	(651) 775-6792	danielm@baywest.com

Table 2-4 Lines of Authority

Name	Organization	Telephone	Cell Number	Email
Jim Hubbell	Bay West Field Operations Lead (FOL)/SSHO	(919) 583-0998	(651) 238-9482	jimh@baywest.com

2.5 Training

Refer to **Section 6.0**, Training in the APP.

2.6 Medical Surveillance

Refer to **Section 9.4**, Medical Surveillance in the APP.

2.7 Exposure Monitoring/Air Sampling Program

Refer to **Section 11.36**, Exposure Monitoring/Air Sampling Program in the APP.

2.8 Personal Protective Equipment

The minimal level of protection required for project personnel and visitors is Level D. The SSHO may increase the level of protection due to changing requirements, but may not decrease the level of protection, without approval of the Safety and Health Manager (SHM) or Certified Industrial Hygienist (CIH). Should personnel encounter an unknown item that may be hazardous, toxic, or radioactive waste (HTRW) they will immediately notify the Project Manager (PM) and evacuate upwind of the suspected item. The PM will notify the United States Army Corps of Engineers (USACE) Point of Contact (POC) of the actions taken.

2.8.1 Level D Protection

Level D Personal Protective Equipment (PPE) provides minimal protection against chemical hazards, and should not be worn in any area with respiratory or skin hazards. A respirator is not required. Level D PPE includes:

- Cotton coveralls or long pants and a shirt with sleeves;
- Hard hat as needed;
- Safety glasses;
- Safety toe work boots with ankle support;
- Work gloves when injury may result from handling materials;
- High visibility vest as needed; and
- Hearing protection, earplugs and/or earmuffs as needed.

2.8.2 Modified Level D Protection

Modified Level D PPE includes the items listed in **Section 2.8.1** above, and a selection of one or more of the following items:

- Tyvek suit, Saranex suit, or Polyvinyl Chloride (PVC) rain suit;
- Safety goggles/face shield;
- Chemical resistant over-boots or chemical resistant safety toe boots;
- Inner nitrile (i.e. 4-mil sample/surgical) gloves;
- Chemical resistant outer gloves; and

- Seal arm, leg, and zipper joints with tape.

2.8.3 Level C Protection

Level C PPE provides a higher level of respiratory and skin protection against chemical hazards. Level C PPE includes the use of an air-purifying respirator. Given the outdoor location of site activities, it is not anticipated that Level C protection will be needed.

2.8.4 Level A and B Protection

Level B PPE should be worn when the highest level of respiratory protection is required, but a lesser level of skin protection is needed. Level A PPE should be worn when the highest level of respiratory and skin protection is needed, or the contaminants of concern are unknown. The activities scheduled for this project will not require the use of Level A or B PPE.

2.8.5 Action Limits

The PPE outlined above was selected according to the site characterization and analysis, job tasks, site hazards, intended use and duration of potential employee exposures.

Given the limited scope of work, implementation of work practice controls and outdoor location, personnel monitoring is not anticipated during site activities. PPE level upgrade/stop will be based on irritation, odor, or olfactory indications of contamination that may be present above PELs or unaccounted for in this SSHP.

Table 2-5 lists the action levels based on monitoring results for these agents:

Table 2-5 Monitoring Action Criteria

Agent	Action Level*	Sampling Duration	Required Action
All	Irritation, odor, or olfactory indications of contamination that may be present above PELs or unaccounted for in this SSHP	More than 1 minute	Evacuate to an upwind location

Notes:
**Monitoring is within the breathing zone of the worker with the highest likely exposure.*

2.9 Heat and Cold Stress

Refer to **Section 11.14**, Heat and Cold Stress Monitoring Plan in the APP.

2.10 SOPs, Engineering Controls, and Work Practices

2.10.1 General Site Rules and Prohibitions

- All site personnel will wear approved head protection, eye/face protection, and foot protection;
- A buddy system is not required for Level D work; however, the PM must account for lone workers;
- Entry into and exit from a site is permitted only through designated access points, except during an emergency, or as authorized by the SSHO;
- Personnel entering a site must wear the required PPE and exit through the personnel decontamination station;

- No eating, drinking, smoking, or any other activity involving hand-to-mouth contact is allowed prior to completing the personnel decontamination sequence. The SSHO will assign the designated break area;
- Facial hair that interferes with a respirator-to-face seal is not be permitted for on-site personnel working in the exclusion zone (EZ) or contamination reduction zone (CRZ) if respiratory protection is required;
- Never enter a confined space (including an excavation or trench) until the SSHO confirms the atmosphere is safe. A permit is required for each permit-required confined space entry;
- All site personnel who wear corrective lenses will provide their own prescription safety glasses and respirator optical inserts;
- Horseplay will not be tolerated;
- Matches, lighters, and any other spark-producing materials are not permitted within 25 feet (ft) of flammables;
- Proper site housekeeping (including removal of trash and orderly stacking and removal of materials to reduce slipping, tripping, and fire hazards) will be the responsibility of all site personnel on a daily basis; and
- Guns and alcohol are not permitted.

2.10.2 Traffic Safety

- Personnel working on-site will obey all posted speed limits. The maximum speed limit is 25 miles per hour (mph) for roads that are not posted. All personnel will be made aware of and abide by speed limits;
- Seatbelts are required for drivers and passengers. The driver will assume responsibility for all passengers wearing seatbelts;
- Proof of insurance will be required for personal vehicles;
- Driving under the influence of alcohol or drugs is prohibited and a violation of company policy and the law;
- Yield to pedestrians;
- Parking is in designated areas only;
- Traffic accidents will be reported immediately to the PM and SHM;
- Cell phone use is not permitted while driving without a hands-free device;
- The vehicle driver will make a walk around inspection prior to engaging vehicle operation to identify any near-by obstructions that may not be apparently visible when sitting in the cab of the vehicle; and
- Vehicles will not be operated in a careless or unsafe manner.

Required equipment features include:

- All vehicles will have an audible backup alarm and an audible warning device (i.e. a horn);
- Each vehicle will have a readily accessible first aid kit; and
- Each vehicle and piece of equipment will have a portable fire extinguisher rated not less than 2-A:10-B:C.

2.10.3 Work Permits

Anticipated work permits include installation dig permits. Work permits will be obtained prior to commencing intrusive work.

2.10.4 Dust Control

Dust control measures are practices to minimize/reduce surface and air movement of dust from disturbed soil surfaces. Dust can be a nuisance to site workers and it may result in adverse health effects by contributing to respiratory health problems, and the environmental impact of dust may be significant. Site activities do not warrant real-time respirable dust measurements; however, if dust is in visible concentrations, one or more of the following dust controls will be implemented:

- Dampen the site slightly during excavation or whenever dust is being raised. Be careful not to wet it to the point that run-off is created;
- Minimize the amount of the site disturbed at any one time;
- Ensure vehicles only enter and exit the site via a stabilized site access route;
- Cover materials and stockpiles;
- Vehicles should be parked where they will not be exposed to dust;
- Provide employees with enclosed equipment cabs;
- Site workers should not allow their clothes to become contaminated with dust so that contaminated material is carried home on their clothes. Other members of the household may then also be exposed;
- Practice good personal hygiene – wash exposed areas prior to eating, drinking, smoking/using tobacco products, using the restroom, applying cosmetics, or exiting the work site;
- Use water to remove dirt/dust from equipment. Do not use compressed air to remove dirt/dust;
- Use spray-on chemical soil treatments (palliatives). Examples of chemical treatments include anionic asphalt emulsion, latex emulsion, resin in water, and natural clays;
- Use mulching as a quick and effective means of dust control for a recently disturbed area;
- Maintain as much vegetation as possible;
- Inspect and sweep roads at the end of each day and when rain is likely;
- Use equipment fitted with dust suppressors. Routinely maintain dust control systems to ensure they are in good working order;
- Install barriers/wind breaks to divert the wind up and over the site (e.g. shade walls of a height that is one-fifth the site length, wind fence, snow fence, tarp curtain, hay bales). Dust collected around barriers should be removed regularly to maintain its effectiveness; and/or
- If the dust becomes a serious problem on windy days (e.g. respirable dust monitor readings exceed 1.5 mg/m^3), stop work until wind conditions are suitable.

2.11 Site Control Measures

2.11.1 Work Zones

The project will be set up based on a modified site zonation system to control the potential spread of contamination. The EZ, CRZ, and Support Zone (SZ) will be identified prior to the start of each task. These zones will be removed once the task is finished, and established at the next work area.

2.11.1.1 Exclusion Zone

The areas which contain, or are suspected to contain, hazardous material are the EZ. Prior to the start of each task, the EZ "hot line" will be clearly identified using caution flagging tape, traffic cones, or other suitable markings. The size of the EZ will depend on the task. All areas that contain, or are suspected to contain, hazardous materials will be considered to be an EZ.

Personnel are not allowed in the EZ without:

- A buddy (not required for Level D work);
- Appropriate PPE;
- Current Occupational Safety and Health Administration (OSHA) medical authorization; and
- Current OSHA training certification.

2.11.1.2 Contamination Reduction Zone

A CRZ will be established between the EZ and the SZ. The CRZ provides personnel, portable equipment, and sampling equipment decontamination. The CRZ will be used for general site entry and exit, and for donning/removing PPE. The CRZ may also contain safety and emergency equipment such as an emergency eyewash, fire extinguisher, and first aid kit. It may also include a heavy equipment decontamination pad, if necessary.

2.11.1.3 Support Zone

The SZ consists of a staging area in a non-hazardous or clean area. It shall contain the team vehicles, a first aid/medical monitoring station, and other elements necessary to support site activities. Normal work clothes and boots may be worn in this area. Location shall be based upon favorable wind direction, topography and site accessibility, when conditions allow.

2.11.2 Site Access and Security

The field team and on-site subcontractor employees will meet any entry requirements of the installation. Personnel not meeting these requirements will not be permitted to work on-site. In general, site control is the responsibility of the SSHO, who will coordinate with the PM on a regular basis.

2.12 On-site Communications

In an emergency, important messages shall be conveyed quickly and accurately. Site personnel shall be able to communicate information even through noise and confusion. Outside support sources shall be reached, assistance obtained, and measures for public notification ensured, if necessary. External communications shall be obtained through cellular phones or radios located in the SZ and procedures shall be posted in an accessible location for all site workers.

Separate internal emergency signals shall be developed and communicated at safety meetings. Verbal communication shall be the primary form of communication at the site. The anticipated

distance between the site workers is no more than 25 ft. Verbal communication at the site can be impeded by on-site background noise and the use of PPE. A vehicle horn or air horn will be available for emergency alerting purposes.

2.13 Personal Hygiene and Decontamination

2.13.1 Contamination Prevention

One important aspect of decontamination is contamination prevention. Good contamination prevention minimizes worker exposure and help to ensure valid sample results by precluding cross-contamination. Procedures for contamination prevention for personnel include:

- Do not walk through areas of obvious or known contamination;
- Do not handle or touch contaminated materials directly. Inspect all PPE to ensure it is free from cuts and tears prior to donning;
- Fasten all closures on suits, covering with tape if necessary;
- Particular care should be taken to protect any skin injuries. If open wounds exist on hands or forearms, handling contaminated materials or samples should be restricted or eliminated;
- Stay upwind of airborne contaminants; and
- Do not carry cigarettes, gum, chewing tobacco, cosmetics, etc. into potentially contaminated areas.

Procedures for contamination prevention for equipment include:

- Limit the amount of contamination that comes in contact with heavy equipment;
- If contaminated tools are to be placed on non-contaminated equipment for transport, use plastic to keep non-contaminated surfaces clean; and
- Keep waste material out of the way of workers.

2.13.2 Personnel Decontamination

Based on the type of task being performed, a personnel decontamination station may be set up at the exit to the EZ. All site personnel exiting the EZ will pass through the decontamination station. To reduce the volume of decontamination water generated, protective clothing will be discarded, instead of cleaned and reused. The generation of decontamination water should be minimized whenever possible.

The following steps will be taken for personnel decontamination when personnel exit the EZ through the CRZ. The decontamination station set-up and procedures below are subject to modification by the SSHO based on actual site conditions after coordination with the SHM.

Table 2-6 Personnel Decontamination Procedure

Step	Task
1.	Deposit all equipment and tools used in the EZ onto plastic sheeting or into plastic-lined containers.
2.	Scrub outer boots and any soiled PPE (i.e. outer gloves, Tyvek) thoroughly with a soapy wash solution and a scrub brush. Rinse off boots and PPE.
3.	Remove tape from around boots and sleeves and dispose of into a plastic-lined trash can.
4.	Remove Tyvek (inside out) and dispose of into a plastic-lined trash can.
5.	Remove outer over-boots and dispose of into a plastic-lined trash can.
6.	Remove outer gloves and dispose of into a plastic-lined trash can.

Table 2-6 Personnel Decontamination Procedure

Step	Task
7.	Remove respirator, remove and discard respirator cartridges, and place in a bucket of respirator sanitizer/cleaner solution. Gently clean with a soft bristle brush, and rinse respirator in warm water. Allow respirator to dry in the SZ.
8.	Remove inner gloves (inside out) and dispose of into a plastic-lined drum.
9.	Proceed to SZ to thoroughly wash face, neck, hands, and forearms prior to eating, drinking, smoking, or using the restroom.

Equipment and supplies needed for the personnel decontamination station may include:

- Plastic buckets for glove wash and rinse;
- Plastic trash can liners;
- Plastic sheeting;
- Wash tubs for boot wash and rinse;
- Detergent/water solution (non-phosphate detergent);
- Respirator sanitizer/cleaner;
- Plastic tubs for respirator wash and rinse;
- Long-handled soft bristle scrub brushes for boot wash;
- Small, soft-bristle scrub brush for respirator wash; and
- 55-gallon drums or trash cans.

Personnel decontamination procedures to be used in the event of an emergency are outlined in **Section 11.2.9** in the APP.

2.13.3 Equipment Decontamination

All equipment and tools will be cleaned prior to site entry to remove grease, oil, dirt, or any other off-site materials. The SSHO will make an inspection of the equipment prior to approving the items for use on-site. The SSHO is responsible for inspecting equipment for adequate decontamination prior to removal off-site.

2.13.4 Disposition of Decontamination Waste

Used PPE will be collected in plastic trash bags and placed in 55-gallon drums or containers. Team personnel and subcontractors will take precautions to prevent contamination from leaving an EZ unless the waste is being transported for disposal.

2.14 Emergency and First Aid Equipment

Refer to **Section 11.2.7**, Emergency and First Aid Equipment in the APP.

2.15 Emergency Response Plan

Refer to **Section 11.2**, Emergency Response Plan (ERP) in the APP.

Refer to **Section 9.0**, Medical Support in the APP.

2.15.1 Off-Site Medical Support

Southern Maryland Hospital Center is the closest hospital to the Site. The distance from the site to the hospital is approximately 6.6 miles, and the total estimated driving time is 15 minutes. A map to the hospital is located as **Appendix 3**.

Appendix 1

Activity Hazard Analysis

1. Mobilization, Demobilization and General Site Activities	
Project Location	Joint Base Andrews, Maryland
Site Location	CS-C503
Contract Number	W9128F-10-D0025-0002
Date Prepared	12/22/2011
Prepared by (Name/Title):	Daniel Musser, Safety and Health Manager
Reviewed by (Name/Title):	Doug Hickey, Certified Industrial Hygienist
Notes: (Field Notes, Review Comments, etc.)	

Overall Risk Assessment Code (RAC) (Use Highest Code): **L**

Risk Assessment Code (RAC) Matrix

Severity	Probability				
	Frequent	**Likely**	**Occasional**	**Seldom**	**Unlikely**
Catastrophic	E	E	H	H	M
Critical	E	H	H	M	L
Marginal	H	M	M	L	L
Negligible	M	L	L	L	L

Step 1: Review each "Hazard" with identified safety "Controls" and determine RAC (see above)

"**Probability**" is the likelihood to cause an incident, near miss, or accident, and identified as: Frequent, Likely, Occasional, Seldom, or Unlikely.

"**Severity**" is the outcome/degree if an incident, near miss, or accident did occur and identified as Catastrophic, Critical, Marginal, and Negligible.

Step 2: Identify the RAC (Probability/Severity) as E, H, M, or L for each "hazard" on AHA. Annotate the overall highest RAC at the top of AHA.

RAC Chart

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Job Steps	Hazards	Controls	RAC
Mobilization / Site Preparation: 1. Establish storage area for equipment; secure materials in storage. 2. Accept deliveries of equipment and site supplies. 3. Stage office trailer, sanitary facilities, equipment, and supplies. 4. Evaluate site access and control; additional measures may be required. 5. Perform visual site inspections. **Demobilization:** 1. Remove equipment. 2. Load unused material for removal off-site. 3. Remove all non-permanent structures.	Vehicle accidents/collisions.	Vehicle operators should look in the direction of travel and look before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy equipment and moving loads. The speed limit is 25 mph on access roads unless otherwise noted. Use defensive driving techniques and limit distractions.	L
	Injury from improper use of hand or power tools.	Only trained personnel will use hand and power tools. Power tools and equipment will be equipped with a shutoff switch. All rotating parts will be properly guarded. Guard against burns from hot equipment.	L
	Electrical shock from energized equipment.	Use GFCI plugs and heavy duty extension cords.	L
	Muscle strain from improper lifting techniques.	Follow proper lifting techniques; no manual lifting of heavy loads.	L
	Excessive noise exposure due to heavy equipment or power tool use.	Provide hearing protection.	L
	Hands/feet caught in pinch points.	Be aware of and keep hands and feet out of potential pinch points; wear heavy work gloves.	L
	Slips, trips, or falls.	Practice good housekeeping procedures by keeping walking and working surfaces free from slip and trip hazards.	L
	Improper storage of flammable and combustible materials causes a fire hazard.	Separate flammable/combustible materials from other materials. Post "No Smoking or Open Flames" signs. Fire extinguishers must be nearby and readily available. Proper hazardous material labeling is required.	L
	Biological hazards (biting or stinging insects, poisonous snakes, wildlife, animal- or insect-borne diseases, poisonous plants).	If insects are a problem, use insect repellant with <30% DEET. Avoid poisonous wildlife. Use barrier creams/ointments as necessary.	L
	Unauthorized personnel.	Maintain positive site control; cease operations if unauthorized entry is made.	L
	Heat and cold stress.	Review signs, symptoms, and prevention techniques. Dress for the weather, in layers of removable clothing. Drink the appropriate fluids on a frequent basis. Enforce buddy system monitoring.	L
	Inclement weather.	Review procedures during tailgate safety meetings.	L

Equipment to be Used	Training Requirements / Competent or Qualified Personnel name(s)	Inspection Requirements

• Motor Vehicles
• Communications Equipment
• First Aid Kit
• Fire Extinguisher
• Eye Wash Bottles
• Level D PPE
  ○ Long or Short Sleeved Shirt and Pants (T-shirts at discretion of SSHO)
  ○ Safety-toe boots
  ○ Safety Glasses
  ○ Hearing Protection (as necessary)
  ○ Hard Hat (as necessary)
  ○ High visibility safety vest (Class II or greater as necessary)
  ○ Work gloves
  ○ If using chain saws and cutting tools include hard hat, face shield (with safety glasses), hearing protection and leg chaps.
• Hand tools
• Digital Camera
• GPS
	Training to be performed by the SSHO unless otherwise specified:
	• OSHA 1910.120 HAZWOPER Program.
	• Hand Tool Safety.
	• Equipment familiarity as required.
	• Knowledge of the Emergency Response and Notifications procedures in accordance with the SSHP.
	• First Aid and CPR training as required by the SSHP.
	• Safe work practices and precautions associated with task being performed in accordance with the Work Plan.
	• Specific response training in accordance with the Work Plan/SSHP.
	• Personnel will meet requirements in accordance with the applicable regulations for the training and use of PPE.
	• Evacuation and emergency procedures in accordance with the SSHP.
	• OSHA qualifications and training as required in accordance with the Work Plan/SSHP.
		Inspections to be performed by the SSHO unless otherwise specified:
		• Daily serviceability check of equipment.
		• Daily communications checks.
		• Daily checks of first aid kits and weekly inventory of kits.
		• Daily check for serviceability, fit, and comfort of PPE.

Activity/Work Task:	**3. Environmental Sampling**
Project Location	Joint Base Andrews, Maryland
Site Location	CS-C503
Contract Number	W9128F-10-D0025-0002
Date Prepared	12/22/2011
Prepared by (Name/Title):	Daniel Musser, Safety and Health Manager
Reviewed by (Name/Title):	Doug Hickey, Certified Industrial Hygienist
Notes: (Field Notes, Review Comments, etc.)	

Overall Risk Assessment Code (RAC) (Use Highest Code): **L**

Risk Assessment Code (RAC) Matrix

Severity	Probability				
	Frequent	**Likely**	**Occasional**	**Seldom**	**Unlikely**
Catastrophic	E	E	H	H	M
Critical	E	H	H	M	L
Marginal	H	M	M	L	L
Negligible	M	L	L	L	L

Step 1: Review each "Hazard" with identified safety "Controls" and determine RAC (see above)

"**Probability**" is the likelihood to cause an incident, near miss, or accident, and identified as: Frequent, Likely, Occasional, Seldom, or Unlikely.

"**Severity**" is the outcome/degree if an incident, near miss, or accident did occur and identified as Catastrophic, Critical, Marginal, and Negligible.

Step 2: Identify the RAC (Probability/Severity) as E, H, M, or L for each "hazard" on AHA. Annotate the overall highest RAC at the top of AHA.

RAC Chart

- E = Extremely High Risk
- H = High Risk
- M = Moderate Risk
- L = Low Risk

Job Steps	Hazards	Controls	RAC
Water Sampling 1. Locate well 2. Unlock well cap 3. Measure depth to water 4. Insert pump or sampling device 5. Cut tubing to length 6. Run electric pumps, generators, or other electrical equipment during well purging/stabilization 7. Refuel generator 8. Purge well 9. Collect groundwater sample 10. Remove purging/ sampling equipment from well 11. Close and lock well cap 12. Decontaminate Equipment 13. Sample Management and Shipping **Soil Sampling** 1. Locate soil sample location 2. GPS 3. Collect sample using Hand Auger / Shovel / Scoop 4. Documentation 5. Collection soil sample	Exposure to site contaminants or vapors.	Set equipment upwind of site work. Evacuate site based on irritation, odor, or olfactory indications of contamination that may be present above PELs or unaccounted for in this SSHP	**L**
	Contact with contaminated material.	Wear the appropriate PPE for the task being performed. Use good personal hygiene: wash hands and face prior to eating, drinking, or tobacco use. Use non-toxic detergent for decontaminated equipment (Alconox). Minimize need for decontamination by using dedicated and/or disposable equipment.	**L**
	Chemical exposure.	Use non-toxic detergent for decontaminated equipment (Alconox). Minimize need for decontamination by using dedicated and/or disposable equipment. Wear appropriate PPE.	**L**
	Severe Weather.	In the event of a tornado warning, severe thunderstorm warning, or the threat of other severe weather conditions, personnel will perform tasks necessary to stabilize the work area and evacuate the site. Personnel will proceed to assigned shelter or assembly points(s) as established during the tailgate safety meeting.	**L**
	Injury from improper use of hand tools.	Only trained personnel will use hand tools.	**L**
	Fire or Explosion.	Generators will be shut down and allowed to cool prior to refueling. Gas cans of 5 gallons or less will be kept in metallic safety UL/FM approved fuel cans.	**L**
	Cut or skin puncture.	Review proper cutting procedures; cut away from your body; use a proper cutting tool; use sharp tools.	**L**
	Unauthorized Personnel.	Maintain positive site control; cease operations if unauthorized entry is made.	**L**
	Muscle strain due to improper lifting.	Rotate the task among workers to share the sample collection duties. Follow proper lifting techniques; no manual lifting of heavy loads. Do not over pack coolers greater than 50 pounds. Use two person lifts for heavy objects.	**L**
	Electrical shock from energized equipment.	Use GFCI plugs and heavy duty extension cords.	**L**
	Slips, trips, or falls.	Practice good housekeeping procedures. Keep walking/working surfaces free from slip/ trip hazards.	**L**
	Biological hazards (biting or stinging insects, poisonous snakes, wildlife, animal- or insect borne diseases).	If insects are a problem, use insect repellant with <30% DEET. Avoid poisonous wildlife. Use barrier creams/ointments as necessary.	**L**

Equipment to be Used	Training Requirements / Competent or Qualified Personnel name(s)	Inspection Requirements
▪ Motor Vehicles ▪ Communications Equipment ▪ First Aid Kit ▪ Fire extinguishers ▪ Eye Wash Bottles ▪ Level D PPE (Modified Level D when working with contaminated soil) ○ Long or Short Sleeved Shirt and Pants (T-shirts at discretion of SSHO) ○ Safety-toe boots ○ Safety Glasses ○ Hearing Protection (as necessary) ○ Hard Hat (as necessary) ○ High visibility safety vest (Class II or greater) ○ Work gloves ○ Nitrile sample gloves when working with contaminated soil ○ Tyvek suits (if necessary) ▪ Hand tools ▪ Sampling Equipment ○ Pump/Controller ○ Generator ○ Tubing ○ Tubing Cutter ○ Buckets/Pails ○ Electrical Cord/GFCI ▪ Stainless steel hand auger ▪ Stainless steel buckets ▪ Disposable trowels ▪ Stainless steel trowels ▪ Stainless steel spoons ▪ Stainless steel mixing bowls ▪ Sample Glassware ▪ Water Level Meter ▪ Digital Camera ▪ GPS	**Training to be performed by the SSHO unless otherwise specified:** • OSHA 1910.120 HAZWOPER Program. • Hand Tool Safety. • Hearing Conservation Program. • Equipment familiarity as required. • Knowledge of the Emergency Response and Notifications procedures in accordance with the SSHP. • First Aid and CPR training as required by the SSHP. • Safe work practices and precautions associated with task being performed in accordance with the Work Plan. • Specific response training in accordance with the Work Plan/SSHP. • Personnel will meet requirements in accordance with the applicable regulations for the training and use of PPE (including respiratory protection). • OSHA qualifications and training as required in accordance with the Work Plan/SSHP.	**Inspections to be performed by the SSHO unless otherwise specified:** • Daily serviceability check of equipment. • Inspect hand tools for excessive wear and loose parts. • Daily communications checks. • Daily checks of first aid kits and weekly inventory of kits. • Daily check for serviceability, fit, and comfort of PPE. • Calibration of purging/sampling equipment.

Appendix 2

Phone Contact List

Table A1 Phone Contact List

Contact	Number
Contact first for any emergency situation	**911**
Medical Emergencies	**911**
Fire Emergencies	**911**
Police Department – Emergency Situations	**911**
Andrews Security – Non-emergency Situations	(301) 981-2001
Andrews Fire Department – Non-Emergency Situations	(301) 981-4985
Site Contact – Lydia Plotz, Joint Base Andrews PM	(678) 478-4917
Emergency Medical Facility – Southern Maryland Hospital Center; 7503 Surratts Road; Clinton, Maryland 20735	(301) 877-4500
Non-emergency Medical Facility –Concentra Medical Center 9141 Alaking Court Suite 112; Capitol Heights, MD 20743	(301) 499-4655
Poison Control Center	(800) 222-1222
Federal OSHA Hotline	(800) 321-6742
State OSHA Hotline	(888) 257-6674
Call Before You Dig	(800) 257-7777; or 811
State EPA – Maryland Department of the Environment	(866) 633-4686
CHEMTREC	(800) 424-9300
CDC - Centers for Disease Control and Prevention	(800) 232-4636
National Response Center	(800) 424-8802
USACE PM – Lucas Walsh	(402) 995-2750 Cell:
Bay West Program Manager – Marty Wangensteen	(651) 291-3475 Cell: (651) 341-3265
Bay West PM – Shirley McMaster	(218) 835-5852 Cell: (651) 341-3263
Bay West Site Lead – Rob Heimbach	(651) 291-3476 Cell: (651) 338-9821
Safety and Health Manager (SHM) – Daniel Musser	(651) 291-3457 Cell: (651) 775-6792
Certified Industrial Hygienist (CIH) – Doug Hickey	(763) 479-3214 Cell: (612) 719-9922
FOL/Site Safety and Health Officer (SSHO) – Jim Hubbell	(651) 238-9482

The SSHO must be notified immediately after calling for emergency assistance or may be called first if the emergency is not life threatening. The SSHO will then notify the PM and SHM. The PM will notify the USACE POC.

Appendix 3

Map to Medical Facility

Figure A1 Map to Medical Facility

Get directions | My places

MD-5 S/Branch Ave **6.6 mi, 15 mins**

I-495/I-95 S and MD-5 S/Branch Ave 8.5 mi, 16 mins

Virginia Ave and MD-5 S/Branch Ave 6.2 mi, 18 mins

Driving directions to Southern Maryland Hospital Center 3D

This route has restricted usage or private roads.

A CS-C503
Arnold Ave

1. Head **south** on **Arnold Ave** toward **Fairbanks St**
Restricted usage road — 0.2 mi

2. Take the 1st right onto **Westover Dr**
Partial restricted usage road — 0.3 mi

3. Turn left onto **Allentown Rd** — 1.6 mi

4. Slight left to merge onto **MD-5 S/Branch Ave** — 4.1 mi

5. Turn left onto **Surratts Rd** — 0.1 mi

6. Turn right toward **Hospital Dr** — 197 ft

7. Continue straight onto **Hospital Dr**
Destination will be on the right — 0.1 mi

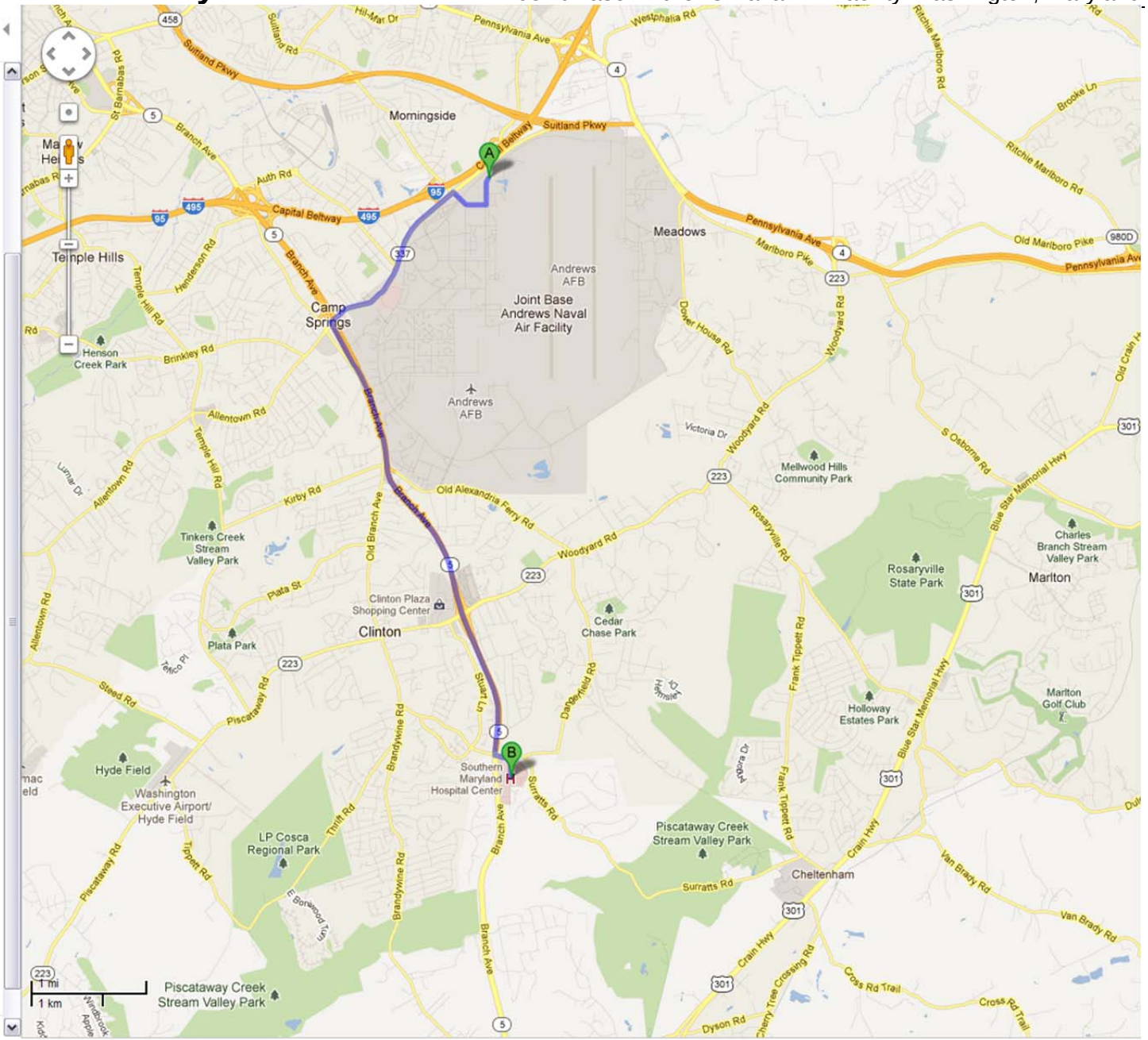
B **Southern Maryland Hospital Center**
9, Surratts, Maryland

Save to My Maps

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan your route accordingly. You must obey all signs or notices regarding your route.

Map data ©2011 Google

Report a problem - Maps Labs - Help



Appendix 4

Material Safety Data Sheets

MATERIAL SAFETY DATA SHEET

Product Trade Name: Bentonite Granular Pack

Revision Date: 04-Mar-2009

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION1. CHEMICAL PRODUCT AND

Product Trade Name: Bentonite Granular Pack
Synonyms: None
Chemical Family: Mineral

Manufacturer/Supplier

TegraSeal Products, LLC
9231 Penn Avenue S, Suite 2A
Bloomington, MN 55431
Telephone: (952) 888-1816
Fax: (952) 888-1786
Emergency Telephone: (508) 816-2168

Prepared By

2. COMPOSITION/INFORMATION ON INGREDIENTS

SUBSTANCE	CAS Number	PERCENT	ACGIH TLV-TWA	OSHA PEL-TWA
Crystalline silica, quartz	14808-60-7	1 - 5%	0.025 mg/m³	10 mg/ m³ %SiO2 + 2
Crystalline silica, cristobalite	14464-46-1	0 - 1%	0.025 mg/ m³	1/2 x 10 mg/ m³ %SiO2 + 2
Crystalline silica, tridymite	15468-32-3	0 - 1%	0.05 mg/ m³	1/2 x 10 mg/ m³ %SiO2 + 2
Bentonite	1302-78-9	60 - 100%	Not applicable	Not applicable

More restrictive exposure limits may be enforced by some states, agencies, or other authorities.

3. HAZARDS IDENTIFICATION

Hazard Overview

CAUTION! - ACUTE HEALTH HAZARD
May cause eye and respiratory irritation.
DANGER! - CHRONIC HEALTH HAZARD
Breathing crystalline silica can cause lung disease, including silicosis and lung cancer. Crystalline silica has also been associated with scleroderma and kidney disease. This product contains quartz, cristobalite, and/or tridymite which may become airborne without a visible cloud. Avoid breathing dust. Avoid creating dusty conditions. Use only with adequate ventilation to keep exposures below recommended exposure limits. Wear a NIOSH certified, European Standard EN 149, or equivalent respirator when using this product. Review the Material Safety Data Sheet (MSDS) for this product, which has been provided to your employer.

4. FIRST AID MEASURES

Inhalation	If inhaled, remove from area to fresh air. Get medical attention if respiratory irritation develops or if breathing becomes difficult.
Skin	Wash with soap and water. Get medical attention if irritation persists.
Eyes	In case of contact, immediately flush eyes with plenty of water for at least 15 minutes and get medical attention if irritation persists.
Ingestion	Under normal conditions, first aid procedures are not required.
Notes to Physician	Treat symptomatically.

5. FIRE FIGHTING MEASURES

Flash Point/Range (F):	Not Determined
Flash Point/Range (C):	Not Determined
Flash Point Method:	Not Determined
Autoignition Temperature (F):	Not Determined
Autoignition Temperature (C):	Not Determined
Flammability Limits in Air - Lower (%):	Not Determined
Flammability Limits in Air - Upper (%):	Not Determined

Fire Extinguishing Media	All standard firefighting media.
Special Exposure Hazards	Not applicable.
Special Protective Equipment for Fire-Fighters	Not applicable.
NFPA Ratings:	Health 0, Flammability 0, Reactivity 0
HMIS Ratings:	Health 0*, Flammability 0, Reactivity 0

6. ACCIDENTAL RELEASE MEASURES

Personal Precautionary Measures	Use appropriate protective equipment. Avoid creating and breathing dust.
Environmental Precautionary Measures	None known.
Procedure for Cleaning / Absorption	Collect using dustless method and hold for appropriate disposal. Consider possible toxic or fire hazards associated with contaminating substances and use appropriate methods for collection, storage and disposal.

7. HANDLING AND STORAGE

Handling Precautions This product contains quartz, cristobalite, and/or tridymite which may become airborne without a visible cloud. Avoid breathing dust. Avoid creating dusty conditions. Use only with adequate ventilation to keep exposure below recommended exposure limits. Wear a NIOSH certified, European Standard En 149, or equivalent respirator when using this product. Material is slippery when wet.

Storage Information Use good housekeeping in storage and work areas to prevent accumulation of dust. Close container when not in use. Do not reuse empty container.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Engineering Controls Use approved industrial ventilation and local exhaust as required to maintain exposures below applicable exposure limits listed in Section 2.

Respiratory Protection Wear a NIOSH certified, European Standard EN 149, or equivalent respirator when using this product.

Hand Protection Normal work gloves.

Skin Protection Wear clothing appropriate for the work environment. Dusty clothing should be laundered before reuse. Use precautionary measures to avoid creating dust when removing or laundering clothing.

Eye Protection Wear safety glasses or goggles to protect against exposure.

Other Precautions None known.

9. PHYSICAL AND CHEMICAL PROPERTIES

Physical State:	Solid
Color:	Various
Odor:	Odorless
pH:	8-10
Specific Gravity @ 20 C (Water=1):	2.65
Density @ 20 C (lbs./gallon):	Not Determined
Bulk Density @ 20 C (lbs/ft³):	50-70
Boiling Point/Range (F):	Not Determined
Boiling Point/Range (C):	Not Determined
Freezing Point/Range (F):	Not Determined
Freezing Point/Range (C):	Not Determined
Vapor Pressure @ 20 C (mmHg):	Not Determined
Vapor Density (Air=1):	Not Determined
Percent Volatiles:	Not Determined
Evaporation Rate (Butyl Acetate=1):	Not Determined
Solubility in Water (g/100ml):	Insoluble
Solubility in Solvents (g/100ml):	Not Determined
VOCs (lbs./gallon):	Not Determined
Viscosity, Dynamic @ 20 C (centipoise):	Not Determined
Viscosity, Kinematic @ 20 C (centistrokes):	Not Determined
Partition Coefficient/n-Octanol/Water:	Not Determined
Molecular Weight (g/mole):	Not Determined

10. STABILITY AND REACTIVITY

Stability Data:	Stable
Hazardous Polymerization:	Will Not Occur
Conditions to Avoid	None anticipated
Incompatibility (Materials to Avoid)	Hydrofluoric acid.
Hazardous Decomposition Products	Amorphous silica may transform at elevated temperatures to tridymite (870 C) or cristobalite (1470 C).
Additional Guidelines	Not Applicable

11. TOXICOLOGICAL INFORMATION

Principle Route of Exposure	Eye or skin contact, inhalation.
Inhalation	Inhaled crystalline silica in the form of quartz or cristobalite from occupational sources is carcinogenic to humans (IARC, Group 1). There is sufficient evidence in experimental animals for the carcinogenicity of tridymite (IARC, Group 2A). Breathing silica dust may cause irritation of the nose, throat, and respiratory passages. Breathing silica dust may not cause noticeable injury or illness even though permanent lung damage may be occurring. Inhalation of dust may also have serious chronic health effects (See "Chronic Effects/Carcinogenicity" subsection below).
Skin Contact	May cause mechanical skin irritation.
Eye Contact	May cause eye irritation.
Ingestion	None known
Aggravated Medical Conditions	Individuals with respiratory disease, including but not limited to asthma and bronchitis, or subject to eye irritation, should not be exposed to quartz dust.

Chronic Effects/ Carcinogenicity

Silicosis: Excessive inhalation of respirable crystalline silica dust may cause a progressive, disabling, and sometimes-fatal lung disease called silicosis. Symptoms include cough, shortness of breath, wheezing, non-specific chest illness, and reduced pulmonary function. This disease is exacerbated by smoking. Individuals with silicosis are predisposed to develop tuberculosis.

Cancer Status: The International Agency for Research on Cancer (IARC) has determined that crystalline silica inhaled in the form of quartz or cristobalite from occupational sources can cause lung cancer in humans (Group 1 - carcinogenic to humans) and has determined that there is sufficient evidence in experimental animals for the carcinogenicity of tridymite (Group 2A - possible carcinogen to humans). Refer to IARC Monograph 68, Silica, Some Silicates and Organic Fibres (June 1997) in conjunction with the use of these minerals. The National Toxicology Program classifies respirable crystalline silica as "Known to be a human carcinogen". Refer to the 9th Report on Carcinogens (2000). The American Conference of Governmental Industrial Hygienists (ACGIH) classifies crystalline silica, quartz, as a suspected human carcinogen (A2).

There is some evidence that breathing respirable crystalline silica or the disease silicosis is associated with an increased incidence of significant disease endpoints such as scleroderma (an immune system disorder manifested by scarring of the lungs, skin, and other internal organs) and kidney disease.

Other Information

For further information consult "Adverse Effects of Crystalline Silica Exposure" published by the American Thoracic Society Medical Section of the American Lung Association, American Journal of Respiratory and Critical Care Medicine, Volume 155, pages 761-768 (1997).

Toxicity Tests

Oral Toxicity: Not determined

Dermal Toxicity: Not determined

Inhalation Toxicity: Not determined

Primary Irritation Effect: Not determined

Carcinogenicity Refer to IARC Monograph 68, Silica, Some Silicates and Organic Fibres (June 1997).

Genotoxicity: Not determined

Reproductive / Developmental Toxicity: Not determined

12. ECOLOGICAL INFORMATION

Mobility (Water/Soil/Air) Not determined
Persistence/Degradability Not determined
Bio-accumulation Not Determined

Ecotoxicological Information

Acute Fish Toxicity: TLM96: 10000 ppm (Oncorhynchus mykiss)

Acute Crustaceans Toxicity: Not determined

Acute Algae Toxicity: Not determined

Chemical Fate Information Not determined

Other Information Not applicable

13. DISPOSAL CONSIDERATIONS

Disposal Method Bury in a licensed landfill according to federal, state, and local regulations.

Contaminated Packaging Follow all applicable national or local regulations.

14. TRANSPORT INFORMATION

Land Transportation

DOT
Not restricted

Canadian TDG
Not restricted

ADR Not restricted

Air Transportation

ICAO/IATA Not restricted

Sea Transportation

IMDG Not restricted

Other Shipping Information

Labels: None

15. REGULATORY INFORMATION

US Regulations

US TSCA Inventory All components listed on inventory.

EPA SARA Title III Extremely Hazardous Substances Not applicable

EPA SARA (311,312) Hazard Class Acute Health Hazard
Chronic Health Hazard

EPA SARA (313) Chemicals This product does not contain a toxic chemical for routine annual "Toxic Chemical Release Reporting" under Section 313 (40 CFR 372).

EPA CERCLA/Superfund Reportable Spill Quantity Not applicable.

EPA RCRA Hazardous Waste Classification	If product becomes a waste, it does NOT meet the criteria of a hazardous waste as defined by the US EPA.
California Proposition 65	The California Proposition 65 regulations apply to this product.
MA Right-to-Know Law	One or more components listed.
NJ Right-to-Know Law	One or more components listed.
PA Right-to-Know Law	One or more components listed.

Canadian Regulations

Canadian DSL Inventory	All components listed on inventory.
WHMIS Hazard Class	Crystalline silica

16. OTHER INFORMATION

The following sections have been revised since the last issue of this MSDS
Not applicable

Additional Information	For additional information on the use of this product, contact your local TegraSeal representative. For questions about the Material Safety Data Sheet for this or other TegraSeal Products, contact 952-888-1816.
Disclaimer Statement	This information is furnished without warranty, expressed or implied, as to accuracy or completeness. The information is obtained from various sources including the manufacturer and other third party sources. The information may not be valid under all conditions nor if this material is used in combination with other materials or in any process. Final determination of suitability of any material is the sole responsibility of the user.

Attachment 2

Construction Quality Plan

FINAL
CONSTRUCTION QUALITY PLAN

PERFORMANCE-BASED RESTORATION
JOINT BASE ANDREWS NAVAL AIR FACILITY WASHINGTON
CAMP SPRINGS, MARYLAND

Contract W9128F-10-D-0025, DO #0002
OCTOBER 2012
VERSION: 01

Prepared for:

U.S. Air Force
11th CES/CEAN
3466 North Carolina Avenue
Joint Base Andrews, Maryland 20762-4803

US Army Corps of Engineers,
Omaha District
1616 Capitol Avenue
Omaha, Nebraska 68102-4901

Prepared by:

Bay West, Inc.
5 Empire Drive
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(651) 291-0456

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ACRONYMS

AMEC ..... AMEC Environment & Infrastructure, Inc.
APP ..... Accident Prevention Plan
Bay West ..... Bay West, Inc.
COR..... Contracting Officer's Representative
CQC..... Contractor Quality Control
CQP..... Construction Quality Plan
DoD ..... Department of Defense
DO ..... Delivery Order
DQCR ..... Daily Quality Control Report
EDD ..... Electronic Data Deliverable
ELAP ..... Environmental Laboratory Accreditation Program
JBA ..... Joint Base Andrews Naval Air Facility Washington
PgM ..... Program Manager
PM ..... Project Manager
PMP..... Project Management Professional
QA ..... Quality Assurance
QA/QC ..... Quality Assurance/Quality Control
QC ..... Quality Control
QSM ..... Quality Systems Manual
SEDD..... Staged Electronic Data Deliverables
SOO..... Statement of Objectives
SSHO ..... Site Safety and Health Officer
UFP-QAPP ..... Uniform Federal Policy Quality Assurance Project Plan
USACE ..... US Army Corps of Engineers
Weston ..... Weston Solutions, Inc.

1.0 INTRODUCTION

This document presents the Site Construction Quality Plan (CQP) for performance-based remediation activities at Joint Base Andrews Naval Air Facility Washington (JBA) located in Camp Springs, Maryland. This plan has been prepared by Bay West, Inc. (Bay West) under Environmental Remediation Services Contract W9128F-10-D-0025, Delivery Order (DO) No. 0002, from the United States Army Corps of Engineers (USACE), Omaha District. This CQP was developed in accordance with the USACE Statement of Objectives (SOO) for Performance-Based Remediation at JBA, Maryland dated 29 April 2011, and the Bay West Corporate Quality Management Plan, dated March 2011.

The objective of the CQP is to establish the project quality control (QC) systems that will ensure project activities are in conformance with project specifications. Bay West is responsible for the QC of work related to the performance of this contract.

Bay West will implement an effective QC system consisting of operational procedures, training, tests, records and a defined QC organization. The CQP will be revised as needed to address additional activities or USACE comments.

Bay West has teamed with AMEC Environment & Infrastructure, Inc. (AMEC) and Weston Solutions, Inc. (Weston) to execute this DO under a Prime/Subcontractor relationship. Subcontracts will be formulated to reflect the detailed scope, performance objectives, specifications, and team contractual and legal responsibilities. Provisions of the basic contract, health and safety, and Quality Assurance (QA)/QC requirements will be followed, as appropriate. Subcontractors may perform work utilizing their own QA controls, provided they are consistent with the quality management program outlined in this CQP and are approved by Bay West.

2.0 ORGANIZATION / RESPONSIBILITIES & AUTHORITIES OF QUALITY CONTROL PERSONNEL

Bay West's policy is to allocate personnel with the appropriate training and authority to develop, refine, and implement our quality systems. A Contractor QC (CQC) Systems Manager and sufficient number of additional qualified personnel have been assigned to the project to ensure contract compliance. A member of the CQC organization will oversee all field activities and will have complete authority to take any action necessary to ensure compliance with the contract. The functional roles and responsibilities related to the project CQC organizational chart (**Figure 2-1**) are presented below.

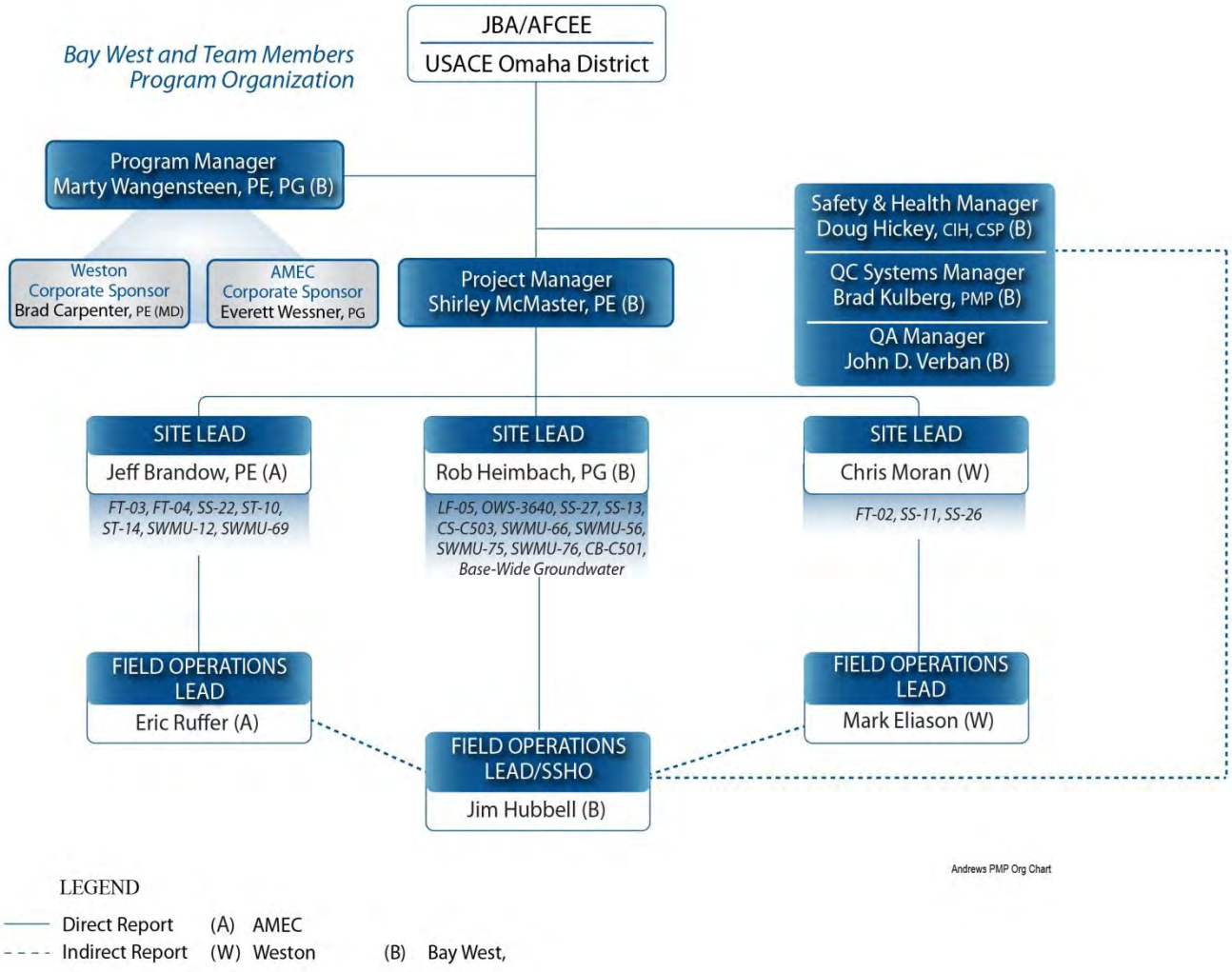


Figure 2-1 Organizational Structure

2.1 Responsibilities and Authorities of the Project CQC Organization

CQC Systems Manager - The Bay West CQC Systems Manager, Brad Kulberg, Project Management Professional (PMP), is responsible for developing, refining, and implementing the Bay West Quality Management Plan and the Chemical QA/QC Plan. The CQC Systems Manager conducts QA performance audits, provides oversight of analytical QA/QC activities, and ensures that training of team members and subcontractors is properly conducted. The CQC Systems Manager remains independent of project line management and ensures enforcement of quality corrective actions. The CQC Systems Manager communicates with the Project Manager to ensure corporate and project QA/QC compliance.

Program Manager (PgM) - The PgM, Martin Wangensteen, is responsible for overall management of this contract and serves as liaison between the contract management team and Bay West's executive team. The PgM is responsible for execution of, and compliance with, all contract requirements, oversight of both health and safety and the QA/QC program, and problem resolution. The PgM works closely with the CQC Systems Manager to integrate QA/QC into all appropriate aspects of the work through all program staff and subcontractors.

Project Manager (PM) - The PM, Shirley McMaster, has overall responsibility for completion of the project in accordance with contract and regulatory requirements. The PM is responsible for the planning and oversight of project activities and acts as an interface between the field staff and corporate office. The PM has the ultimate responsibility for the implementation of the project tasks, safety and health of project works, and quality of the work performed. The PM is responsible for ensuring the review of submittals, maintenance of records, and ensuring that personnel receive the appropriate training and qualifications for the tasks performed.

QA Manager - The QA Manager, John Verban, is responsible for overseeing the performance of off-site analyses and QA/QC in accordance with the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP). This includes the verification that all laboratories utilized for the project are Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accredited and are compliant with the DoD Quality Systems Manual (QSM). The QA Manager is responsible for verifying proper sample containers, shipment methods, complete chain-of-custody paperwork, and laboratory reports including the analytical sample results. The QA Manager is also responsible for coordinating the transfer of electronic data deliverables (EDDs) from the laboratory, data validation, final project reports, and that all reports shall include Staged Electronic Data Deliverables (SEDD) Version 5.0, Stage 2a files, Air Force Center for Engineering and the Environment Environmental Resources Program Information Management System EDD compatible with ERPToolsX 5.0, and an Environmental Quality Information System compatible EDD as specified by Bay West.

Safety and Health Manager – The Safety and Health Manager, Doug Hickey, establishes the health and safety program and procedures for task order execution. The Safety and Health Manager supports the PM, Field Operations Lead, and SSHO in evaluating site controls, practices, and personal protective equipment (PPE). The Accident Prevention Plan and health and safety related corrective actions are developed and/or approved by the Health and Safety Manager.

Site Lead – The Site Leads, Rob Heimbach of Bay West, Jeff Brandow of AMEC and Chris Moran of Weston, area responsible for planning and leading work execution associated with assigned sites to accomplish objectives, and also communicate with the PM to provide scope schedule and cost information. Site Leads review and approve site-specific technical reports and documents and manage individual site subcontractors.

Field Operations Lead – The Bay West Team Field Operations Lead (FOL), Jim Hubbell, is responsible for the completion of site operations in accordance with approved plans and procedures. The FOL or designee will be on-site at all times during construction. The FOL has the authority to act on behalf of the Site Lead and can stop work due to on-site-related issues affecting the quality of the work performed. In the event of his absence, a qualified individual will be appointed to serve as his replacement. The requirements for the alternate will be the same as for the designated FOL. The Bay West FOL is the primary FOL and will oversee and coordinate with the AMEC and Weston FOLs.

Site Safety and Health Officer (SSHO) – The Bay West Team SSHO is responsible for the implementation of the Accident Prevention Plan (APP) and directs task order personnel and sub-contractors on safety-related issues. The SSHO, or his designee, has the authorization to stop work and to ensure corrective action for noncompliance with the APP.

3.0 SUBMITTALS

The Bay West Team will comply with submittal procedures specified in the USACE SOO. These include procedures for scheduling, reviewing, certifying, and managing submittals, including those of subcontractors, suppliers, and purchasing agents. Bay West is responsible for reviewing specifications, contract drawings, plans, and reports. A list of the minimum document submittals is provided in **Table 3-1**.

Table 3-1 Minimum Document Submittals

Document	USAF/ USACE Review	Regulatory Review
Project Management Plan	X	
Site Specific UFP-QAPPs	X	X
Other Site Specific Technical Reports	X	X
Proposed Plans	X	X
Records of Decision	X	X
Contractor Progress, Status and Management Reports	X	
Meeting Notes	X	X*

*: As applicable

Site-specific UFP-QAPP submittals will be made in advance of commencement of work to allow sufficient time for review and comment. Bay West is responsible for ensuring that all submittals are in compliance with the contract requirements. Other site-specific technical reports will be prepared to document findings of the Site phases of work. Proposed Plans and Records of Decision will summarize remedy selection and document remedial action plans, respectively, for the Sites.

Bay West will maintain a complete, up-to-date file of all submittals.

Submittal Control

All submittals will be delivered to the following addresses:

Lucas Walsh, Project Manager
CENWO-PM-HB
Army Corps of Engineers, Omaha District
1616 Capitol Avenue
Omaha, NE 68102-4901

David Connolly, ERP Interim Chief
Interim Chief, Environmental Restoration Program
11 CES/CEAN
3466 N. Carolina Ave.
Joint Base Andrews, MD 20762

The Bay West PM will coordinate with USACE and JBA to determine the distribution and number of additional copies that will be required. All documents will be produced with working copy, draft and final versions. The Bay West Team will respond to comments and promptly furnish a corrected submittal in the format and number of copies specified by the USACE, JBA, and the other stakeholders.

All documents will be identified as working copy until acceptance of the responses to comments, at which time they will become draft. Similarly, draft versions of report will remain as draft until acceptance of the responses at which time they will become final. Submittal of draft and final documents to USACE, JBA and regulatory agencies shall be performed by Bay West under USACE direction. All submittals will include a cover letter which indicates the project, project phase, the date comments are due and to whom comments are to be submitted, and the date and location of the review comments, etc., as appropriate.

Disapproved Submittals

The Bay West Team will make all corrections required by the Contracting Officer's Representative (COR) and promptly furnish a corrected submittal. If Bay West considers any correction indicated on the submittals to constitute a change to the contract, then written notice, as required under the contract clause entitled "Changes," shall be given to the COR.

4.0 CONTROL, VERIFICATION AND ACCEPTANCE TESTING PROCEDURES

This section includes the control, verification, and acceptance testing procedures for each specific test to be performed including the test name, test frequency, and person responsible for each test.

Required Tests

Environmental testing and analysis has not been included in this CQP. Environmental testing and analysis is included in the site-specific UFP-QAPP prepared in advance of field mobilization for the associated work. Reporting of laboratory test results is also included in each site-specific UFP-QAPP.

5.0 QUALITY CONTROL METHODS

Contractor QC is the means by which the Contractor ensures that the work, to include that of subcontractors and suppliers, complies with the requirements of the contract for each definable feature of the remedial action work.

The Bay West Team will perform three phases of control for each definable feature of work. The three phases of control include Preparatory, Initial, and Follow-Up. Preparatory Phase control will be used to establish quality prior to mobilization and commencement of site activities and delivery of materials. Initial Phase control will verify that all necessary procedures have been instituted to ensure conformance with the project plans. Follow-Up Phase control will include daily checks and documentation of contract requirements to ensure that quality work will be produced throughout the duration of the project.

Preparatory Phase

This phase will be performed prior to beginning work on each definable feature of the work, after the Site-Specific UFP-QAPP is approved. Work cannot begin on a definable feature of work until the Preparatory Phase is complete. The Preparatory Inspection Checklist is included in **Appendix 1**. The following activities will also be performed during the Preparatory Phase:

- Review of contract specifications and drawings;
- Review of regulations;
- Identification of project team;
- Identification and completion of training;
- Verification that all materials and/or equipment have been tested, submitted, and approved;
- Review of provisions made to provide required control inspections and testing;
- Physical check of materials and equipment to confirm they are on-site and conform to project requirements;
- Examination of the work area(s) to assure that all preliminary work has been completed and is in compliance with the contract;
- Review of the appropriate activity hazard analysis to assure safety requirements are met;
- Discussion of procedures for controlling the quality of the work, including repetitive deficiencies. Documentation of construction tolerances and workmanship standards for each feature of work; and
- Confirmation that the portion of the plan for the work to be performed has been accepted by the COR.

Initial Phase

The Initial Phase is defined as the beginning of a definable feature of work. The initial checklist is included in **Appendix 1**. The following will be accomplished during the Initial Phase:

- Check the work to ensure that it is in full compliance with contract requirements, including the review of the preparatory meeting minutes;
- Verify the adequacy of controls to ensure full contract compliance and verify the required control inspection and testing;
- Establish the level of workmanship and verify that it meets minimum acceptable workmanship standards;
- Resolve all differences;
- Check safety activities to include compliance with and upgrading of the Site Safety and Health Plan and activity hazard analysis. Review the activity hazard analysis with each worker and document the review on the daily report; and

- Repeat the Initial Phase for each new crew member on-site, or any time acceptable specified quality standards are not being met. Document all orientations.

Additional Preparatory and Initial Phases

Additional Preparatory and Initial Phases will be conducted on the same definable features of work if the quality of on-going work is unacceptable; if there are changes in the applicable CQC staff, on-site production supervision, or work crew; if work on a definable feature is resumed after a substantial period of inactivity; or if other problems develop.

Follow-Up Phase

Daily checks will be performed to assure control activities are providing continued compliance with contract requirements, until completion of the particular feature of work. The checks will be recorded on the Daily Quality Control Report (DQCR). An example is included in **Appendix 1**. Final follow-up checks shall be conducted and all deficiencies corrected prior to the start of additional features of work that may be affected by the deficient work. The Bay West Team will not build upon nor conceal non-conforming work.

Documentation

The Bay West Team will maintain current records providing factual evidence that the required QC activities have been performed. These records will include the work of subcontractors and suppliers. Project documentation will be maintained for a minimum of five years. Items to be documented include, but are not limited to, the following:

- Field readiness review checklist;
- Project kickoff checklist;
- Instrument calibration and maintenance records;
- Field notebooks and daily activity logs;
- Sample collection logs;
- Field monitoring/screening results and associated data sheets;
- Equipment inspection checklists;
- Sample labels and chain-of-custody records;
- Training records;
- Pre-entry health and safety briefings and daily tailgate safety meetings; and
- DQCRs.

The DQCR includes, as a minimum, the following information:

- Contractor/subcontractors on-site and their area of responsibility;
- Equipment on-site with hours worked, idle, or down for repair;
- Work performed each day, giving location, description, and by whom;
- QC activities performed (including field calibrations);
- Health and safety levels and activities; and
- Problems encountered/corrective actions taken.

DQCRs will indicate a description of the field activities performed (investigations, construction, excavations, remediation system operational checks, and regular long-term monitoring/long-term operations events), the number of personnel working, weather conditions, and any delays encountered. These records will cover both conforming and deficient conditions. All calendar days during the site-specific fieldwork will be accounted for. As a minimum, one report will be prepared and submitted to account for each time period when no fieldwork is being performed. Reports will be signed and dated by the FOL.

6.0 COMPLETION INSPECTIONS

This section describes the procedures for tracking construction deficiencies from identification through acceptable corrective action. It also defines the verification procedures which establish that the identified deficiencies have been corrected.

Punch-Out Inspection

The FOL will inspect the work near the completion of all work or any increment thereof established by a completion time stated in the Special Clause entitled, "Commencement, Prosecution, and Completion of Work," or stated elsewhere in the specifications. The FOL will develop a punch list of items that do not conform to the approved drawings and specifications. The punch list of deficiencies will be included in the CQC documentation and will include the estimated date by which the deficiencies will be corrected.

The FOL or staff shall make a second inspection to ascertain that all deficiencies have been corrected. Once this is accomplished, the Bay West Team will notify the COR that the site is ready for the USACE Pre-Final inspection.

Pre-Final Inspection

The USACE will perform this inspection to verify that the work is complete. A USACE Pre-Final Punch List may be developed as a result of this inspection. The FOL will ensure that all items on this list have been corrected before notifying the USACE that a Final Inspection can be scheduled. Any items noted on the Pre-Final Inspection will be corrected in a timely manner. These inspections and any deficiency corrections required by this paragraph shall be accomplished within the time slated for completion of the entire work or any particular increment thereof, if the project is divided into increments by separate completion dates.

Final Acceptance Inspection

The FOL and the COR will be in attendance at this inspection. Additional USACE personnel may also be in attendance. The Final Acceptance Inspection will be formally scheduled by the COR based upon results of the Pre-Final inspection.

Fourteen (14) days notice shall be given to the COR prior to the Final Acceptance Inspection and will include the Bay West Team's assurance that all specific items previously identified as being unacceptable, along with all remaining work performed under the contract, will be complete and acceptable by the date scheduled for the final acceptance inspection.

Notification of Noncompliance

The COR will notify Bay West of any detected noncompliance with the foregoing requirements. Bay West will take immediate corrective action after receipt of such notice. Such notice, when delivered to the Bay West Team at the work site, shall be deemed sufficient for the purpose of notification. If the Bay West Team fails or refuses to comply promptly, the COR may issue an order stopping all or part of the work until satisfactory corrective action has been taken.

Quality Assurance Comments

During the course of the contract, Bay West may receive various QA comments from USACE that will reflect site activity corrections or reflect outstanding or future items needing attention. The Bay West Team will acknowledge receipt of these comments by specific number reference on the DQCR, and the DQCR shall also reflect when these items are specifically completed or corrected to permit USACE verification.

7.0 ASSIGNMENT LETTER

The letter delegating QC authority and responsibility to Brad Kulberg, Bay West's CQC Systems Manager and Jim Hubbell, Bay West's Field Operations Lead, is contained in **Appendix 2**.

Appendix 1

Forms

DAILY CONSTRUCTION QUALITY CONTROL REPORT

Contract No.: W9128F-10-D-0025, DO # 0002 **Date:** _____ **Rpt. No.:** ___

Project Title & Location: _____

Weather: ☐ Clear ☐ Partly Cloudy ☐ Cloudy ☐ Rainfall (% of workday)
Temperature during workday: High °F. Low °F.

1. WORK PERFORMED BY CONTRACTORS/SUBCONTRACTORS

Contractor	No. of Workers	Crafts	Hours	Description of Work

2. OPERATING EQUIPMENT DATA (NOT HAND TOOLS)

Equipment	Date of arrival /departure	Owned or Rented	Hours Used	Hours Idle	Hours of Rep./Main

3. WORK PERFORMED TODAY (Indicate location and description of work performed by prime and/or subcontractors):

4. QUALITY CONTROL INSPECTIONS & RESULTS (Include a description of preparatory, initial, and/or follow-up inspections or meetings; check of subcontractors work and materials delivered to site compared to submittals and/or specifications; comments on proper storage of materials; include comments on corrective actions to be taken):

5. QUALITY CONTROL TESTING AND RESULTS (comment on tests and attach test reports):

DAILY CONSTRUCTION QUALITY CONTROL REPORT

6. DAILY SAFETY INSPECTIONS (Include comments on new hazards to be added to Hazard Analysis and corrective action of any safety issues):
7. REMARKS (Include conversations with or instructions from the Government representatives; delays of any kind that are affecting the job; conflicts in the contract documents; comments on change orders; environmental considerations; etc.):

8. CONTRACTOR'S VERIFICATION: I certify that to the best of my knowledge the above report is complete and correct. All material, equipment used, and work performed during this reporting period is in compliance with the contract plans and specifications except as noted above.

_____

Contractor Quality Control Officer

Initial Inspection Checklist

CONTRACT NO: W9128F-10-D-0025, DO # 0002 **DATE:**

TITLE: Joint Base Andrews, Maryland – Performance-Based Restoration

DESCRIPTION OF WORK INSPECTED:

A. PERSONNEL PRESENT:

	NAME	POSITION	COMPANY
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

B. MATERIALS BEING USED ARE IN STRICT COMPLIANCE WITH THE CONTRACT SPECIFICATIONS:

Yes ☐ No ☐

IF NOT, EXPLAIN:

Initial Inspection Checklist

C. **PROCEDURES AND/OR WORK METHODS WITNESSED ARE IN STRICT COMPLIANCE WITH THE REQUIREMENTS OF THE CONTRACT SPECIFICATIONS:**

Yes ☐ No ☐

IF NOT, EXPLAIN:

D. **WORKMANSHIP IS ACCEPTABLE:**

Yes ☐ No ☐

STATE AREAS WHERE IMPROVEMENT IS NEEDED:

E. **SAFETY VIOLATIONS AND CORRECTIVE ACTION TAKEN:**

_____

QUALITY CONTROL REPRESENTATIVE

Preparatory Inspection Checklist

CONTRACT NO: W9128F-10-D-0025, DO # 0002 **DATE:**

TITLE: Joint Base Andrews, Maryland – Performance-Based Restoration

MAJOR DEFINABLE SEGMENT OF WORK:

A. PERSONNEL PRESENT:

	NAME	POSITION	COMPANY
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

B. ITEMS INVOLVED:

	ITEM	SPECIFICATION OR PLAN SECTION	CONTRACTOR OR GOVERNMENT APPROVAL
1.			
2.			
3.			
4.			
5.			
6.			

Preparatory Inspection Checklist

7. _____ _____ _____
8. _____ _____ _____
9. _____ _____ _____
10. _____ _____ _____
11. _____ _____ _____
12. _____ _____ _____
13. _____ _____ _____
14. _____ _____ _____
15. _____ _____ _____

B-I. HAVE ALL ITEMS INVOLVED BEEN APPROVED?

Yes ☐ No ☐

B-II. WHAT ITEMS HAVE NOT BEEN APPROVED?

1. _____
2. _____
3. _____
4. _____
5. _____

C. TESTS REQUIRED IN ACCORDANCE WITH CONTRACT REQUIREMENTS:

	TEST	**SPECIFICATION OR PLAN SECTION**
1.		
2.		
3.		
4.		
5.		

Preparatory Inspection Checklist

D. ACCIDENT PREVENTION PREPLANNING - HAZARD CONTROL MEASURES:

Applicable HASP Sections

1. _____
2. _____
3. _____
4. _____
5. _____

_____
QC Manager

Appendix 2

Delegation Letter

Bay West
Customer-Focused Environmental & Industrial Solutions

Bay West Inc.
5 Empire Drive, St. Paul, MN 55103
651-291-0456 * FAX 651-291-0099
www.baywest.com * info@baywest.com

December 9, 2011

Re: Delegation of Quality Control Authority

To whom it may concern:

This letter appoints Mr. Brad Kulberg as Bay West's Contractor Quality Control (CQC) System Manager and Mr. Jim Hubbell as Bay West's Field Operations Lead (FOL). This letter hereby provides the CQC Systems Manager and FOL with the responsibility to perform any functions which are required to effectively maintain a quality control program, as described in Bay West's Contractor Quality Control Plan (CQCP) prepared for the Performance-Based Restoration Joint Base Andrews Naval Air Facility Washington, Camp Springs, Maryland (Contract: W9128F-10-D-0025, DO#0002).

The CQC process will include the three phases of control (preparatory, initial, and follow-up phases) as discussed in the CQCP. The CQC Systems Manager and FOL will review the elements of the CQCP for specific instructions regarding these control phases. The CQC Systems Manager and FOL are also directed to review and become familiar with the contract specifications, amendments, and drawings in their entirety. If any part of these documents is not understood, please consult me.

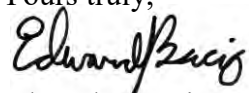
The FOL will be present during all on-site testing and will coordinate all such tests with the USACE's Contracting Officer's Representative (COR) as required by the contract specifications. In the absence of the FOL, an authorized representative, as approved by the CQC Systems Manager, will be assigned to perform the CQC functions.

The FOL will make, on a continuing basis, sufficient daily follow-ups to ensure that all workmanship and materials for this project are in conformance with the contract specifications and drawings.

All control phases and tests will be recorded on the Daily Construction Quality Control Report. These reports, along with all test results, will be reviewed and approved by the FOL or authorized representative prior to being transmitted to the COR.

This delegation includes the authority to stop work that does not comply with the contract.

Yours truly,



Edward J. Bacig
Vice President

BWJ110202; DMS #1460977